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SELECTIVE EXTRACTION OF NITROGEN COMPONENTS FROM FLUID CATALYTIC CRACKING FEEDSTOCKS

Final Report

by

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TABLE OF CONTENTS

		Page No.
	EXECUTIVE SUMMARY	i
1.	INTRODUCTION	1
	1.1 Objectives	1
	1.2 Justification	1
	1.3 Background	2
2.	EXPERIMENTAL	3
	2.1 Preparation of Feedstocks	3
	2.2 Static Extraction Procedure	4
	2.3 Micro Activity Testing	4
	2.4 Static Extractions with Alternate Aprotic Dipolar Solvents	5
	2.5 Optimization of Water Content and Extraction Ratios for Dimethyl Sulfoxide Solvent Systems	6
	2.6 Multistage Extractions with DMSO/H_2O (94:6)	6
	2.7 2nd Stage DMSO Extractions with Aqueous 0.2 and 2.0N p-Toluene Sulphonic Acid	6
	2.8 Relationship of Temperature - Nitrogen Removal Upon Optimum DMSO Solvent System	6
	2.9 Determination of Aprotic Solvents in Raffinates by High Pressure Liquid Chromatography	7
	2.10 Characterization of Extracts	7
3.	DISCUSSION OF RESULTS	8
	3.1 Extractions with Alternate Aprotic Dipolar Solvents	9
	3.2 Optimum Extractions with DMSO Solvent Systems and Multistage Extractions	9
	3.3 2nd Stage DMSO Extractions with Aqueous 0.2 and 2.0N p-Toluene Sulphonic Acid	11
	3.4 Analyses of Aprotic Solvent Remaining in Gas Oil Raffinates	11
	3.5 Effect of DMSO Solvent Upon the Micro-activity Test	12
	3.6 Compositional Characterization of Extracts	12
4.	SUMMARY OF RESULTS	13
5.	ACKNOWLEDGEMENTS	15
6.	REFERENCES	16
7.	TABLES AND FIGURES	
	APPENDIX	

LIST OF TABLES

TABLE 1	GAS OIL FEED DISTILLATIONS
TABLE 2	GAS OIL FEED CHARACTERIZATIONS
TABLE 3	MICRO ACTIVITY TEST RESULTS - CANMET GAS OIL AND DMSO RAFFINATE VARIATION IN CATALYST TO GAS OIL RATIOS
TABLE 4	MICRO ACTIVITY RESULTS - SUNCOR GAS OIL AND DMSO RAFFINATE VARIATION IN CATALYST TO GAS OIL RATIO DATA
TABLE 5	MICRO ACTIVITY TEST RESULTS - SYNCRUDE GAS OIL FEED AND DMSO RAFFINATE VARIATION IN CATALYST TO GAS OIL FEED RATIO DATA
TABLE 6	MICRO ACTIVITY TEST RESULTS - GAS OIL DUPLICATION DATA
TABLE 7	MICRO ACTIVITY TEST RESULTS - DMSO/H_2O (94:6) RAFFINATE DUPLICATION DATA
TABLE 8	COMPARISON OF SOLVENT SYSTEMS NITROGEN REMOVAL CAPABILITIES
TABLE 9	MICRO ACTIVITY TEST RESULTS - COMPARISON OF SOLVENT SYSTEMS NITROGEN REMOVAL CAPABILITIES
TABLE 10	CANMET GAS OIL - DIMETHYL SULFOXIDE EXTRACTIONS
TABLE 11	SUNCOR GAS OIL - DIMETHYL SULFOXIDE EXTRACTIONS
TABLE 12	SYNCRUDE GAS OIL - DIMETHYL SULFOXIDE EXTRACTIONS
TABLE 13	CANMET GAS OIL - DMSO EXTRACTIONS - M.S. ANALYSES
TABLE 14	SUNCOR GAS OIL - DMSO EXTRACTIONS - M.S. ANALYSES
TABLE 15	SYNCRUDE GAS OIL - DMSO EXTRACTIONS - M.S. ANALYSES
TABLE 16	MICRO ACTIVITY TEST RESULTS - VARIATION IN DMSO SOLVENT TO GAS OIL EXTRACTION RATIO
TABLE 17	CANMET AND SYNCRUDE DMSO CROSS-CURRENT EXTRACTIONS
TABLE 18	CANMET AND SYNCRUDE GAS OIL - DMSO/H_2O (94:6) NITROGEN CONTENT EQUILIBRIUM DATA
TABLE 19	MICRO ACTIVITY TESTS RESULTS - CANMET CROSS-CURRENT MULTISTAGE DMSO EXTRACTIONS
TABLE 20	MICRO ACTIVITY TEST RESULTS - SYNCRUDE CROSS-CURRENT MULTISTAGE DMSO EXTRACTIONS
TABLE 21	2ND STAGE DMSO + p-TOLUENE SULPHONIC ACID EXTRACTIONS
TABLE 22	MICRO ACTIVITY TEST RESULTS - DMSO + p-TOLUENE SULPHONIC ACID EXTRACTIONS
TABLE 23	DMSO/H_2O (94:6) TEMPERATURE EXTRACTION DATA
TABLE 24	MICRO ACTIVITY TEST RESULTS - COMPARISON OF CANMET AND SYNCRUDE DMSO/H_2O (94:6) RAFFINATES WITH VARIATION IN EXTRACTION TEMPERATURE
TABLE 25	HPLC APROTIC SOLVENT ANALYSIS OF GAS OIL RAFFINATES
TABLE 26	MICRO ACTIVITY TEST RESULTS - DMSO CONTAMINATED RAFFINATES (1% DMSO)
TABLE 27	COMPOSITIONAL CHARACTERIZATION OF EXTRACTS
TABLE 28	GLOSSARY FOR FIGURES 6, 7 AND 8

LIST OF FIGURES

FIGURE 1 TYPICAL ASSEMBLY WITH GLASS STILL (DEAN AND STARK)

FIGURE 2 MICRO ACTIVITY TEST UNIT

FIGURE 3 CANMET GAS OIL EQUILIBRIUM RELATIONSHIP

FIGURE 4 SYNCRUDE GAS OIL EQUILIBRIUM RELATIONSHIP

FIGURE 5 CRACKABILITY OF SYNTHETIC GAS OILS

FIGURE 6 MAT PRODUCT YIELDS - CANMET GAS OIL RAFFINATES

FIGURE 7 MAT PRODUCT YIELDS - SUNCOR GAS OIL RAFFINATES

FIGURE 8 MAT PRODUCT YIELDS - SYNCRUDE GAS OIL RAFFINATES

EXECUTIVE SUMMARY

Selective extractions employing dipolar aprotic solvent systems such as N,N-Dimethyl Formamide/Water (90:10), N-Methyl 2-Pyrrolidone/Water (90:10) and Dimethyl Sulfoxide/Water (94:6) will partially denitrogenate gas oil fractions derived from either conventional or synthetic sources.

Optimum conditions for maximizing nitrogen removal for Dimethyl Sulfoxide solvent systems are as follows:

Solvent Water Content	= 6% by weight
Solvent to Gas Oil Extraction Ratio	= 1/1 by volume
Extraction Temperature	= 100°C

The resulting partially denitrogenated gas oil raffinate possesses superior FCC processing properties i.e. a substantial increase in the C_5^+ gasoline product and C_3-C_4 hydrocarbon yields can be expected from FCC processing of this type of feedstock.

SOMMAIRE-RECOMMANDATION

Il est possible de déazoter partiellement des fractions de gazole (dérivées de sources classiques ou synthétiques) à l'aide d'extractions sélectives faisant appel à des systèmes de solvants aprotiques bipolaires tels que le N,N-diméthylformamide et l'eau (90:10), le N-méthylpyrrolidone-2 et l'eau (90:10) et le diméthylsulfoxyde et l'eau (94:6).

Dan le cas du système de solvant au diméthylsulfoxyde, les conditions optimales pour enlever le maximum d'azote sont les suivantes:

Teneur en eau du solvant = 6% en poids

Ratio d'extraction solvant-gazole = 1/1 en volume

Température d'extraction = 100°C

Le raffinat de gazole partiellement déazoté résultant de cette extraction possède de meilleures propriétés pour le raffinage par CCF que les fractions non traitées. Ce type de charge d'alimentation permet en effet une augmentation substantielle des quantités d'essence C_5+ et d'hydrocarbures C_3-C_4 produites.

1. INTRODUCTION

1.1 Objectives

The objectives of this project are as follows:

- To optimally remove by aprotic dipolar solvent extraction undesirable FCC compounds such as nitrogen and related complex polar aromatic components from high boiling synthetic gas oil fractions derived from CANMET, Suncor and Syncrude sources.

- To measure the anticipated improvement in FCC product yield resulting from the reduction of these undesirable components in the gas oil feed by Micro Activity Testing (MAT).

- To classify the chemical nature of the components removed by solvent extraction from the three synthetic gas oil fractions.

- To develop an analytical method for monitoring the level of aprotic solvent remaining in the extracted FCC feedstocks.

1.2 Justification

When synthetic crude oils become more economically attractive, their use will be primarily in the production of liquid transportation fuels. Currently, a major portion of gasoline and diesel blending components are derived from FCC processing of gas oil fractions obtained from conventional crude oil sources.

Improved FCC processing conditions such as the reduction of gas oil total nitrogen content is expected to both accelerate the application of synthetic crudes and assist in improved processing of conventional gas oil fractions.

Initially, Contract 15SQ.23440-3-9057 (1) demonstrated the use of aprotic dipolar solvents to selectively remove nitrogen species from FCC feedstocks and improve light liquid product yields. This project was designed to compliment the earlier work and to optimize solvent composition and extraction conditions relating to maximum nitrogen content removal and to study the subsequent improved FCC processability for high boiling gas oil fractions primarily derived from CANMET, Suncor and Syncrude sources.

1.3 Background

Background information relating to this subject has been previously reported in two earlier studies, EMR File No. 15SQ.23440-3-9057 (1) "Impact of Problem Components on Synthetic Crude Processing" and EMR File No. 23SQ.23440-4-9111 (2) "The Compositional Study of Nitrogen and Oxygen Compounds in Products of Heavy Oil Primary and Secondary Upgrading Processes".

2. EXPERIMENTAL

2.1 Preparation of Feedstocks

Raw Suncor and Syncrude gas oils were cut at 360°C using a 22-L atmospheric distillation apparatus. CANMET reactor product was cut into a IBP-200°C naphtha, 200-315°C middle distillate, 315-524°C gas oil and a 524°C+ residuum fraction. The CANMET naphtha, middle distillate and residuum fractions were returned to the EMR CANMET Laboratories, while the gas oil fraction was retained for this study.

Details of the CANMET, Suncor and Syncrude distillations are given in Table 1. The associated product inspection data is given in Table 2.

Previously, two propane deasphalted oils (PDA) were also prepared as feedstocks. Details of this feedstock preparation and associated product quality testing is given in the appendum of this report.

Although seven gas oil feedstocks were prepared in this study i.e. CANMET 315-524°C, Suncor IBP-360°C, Suncor 360°C+, Syncrude IBP-360°C, Syncrude 360°C+, a severely propane deasphalted oil (PDA) and a mildly propane deasphalted oil, this study was basically confined to three of these gas oil fractions. The three gas oil fractions studied were CANMET 315-525°C, Suncor 360°C+ and Syncrude 360°C+.

As the PDA oils were essentially solids up to approximately 80°C, earlier work (see Appendum B) encountered difficulties in water washing the remaining aprotic solvent from the extracted PDA oils due to the stability of the hydrocarbon-water emulsions and the solid waxy nature of the feedstock. Attempts to remove the water residue often resulted in severe bumping and raffinate loss. In some cases, nitrogen analyses on seemingly successfully extracted PDA oils showed an increase in nitrogen content indicating in some cases a substantial amount of aprotic nitrogen containing solvent remained in the feedstock.

2.2 Static Extraction Procedure

Due to difficulties in separating gas oil, water, aprotic solvent and associated emulsions, the following extraction procedure was used in an effort to obtain realistic percent raffinate recovery values:

- 100 g of gas oil was weighed into a tared 500 mL bottle.
- An equal volume of extraction solvent was added and the mixture was agitated for 1 hour with a mechanical shaker.
- The mixture was then transferred to a 500 mL separatory funnel and following overnight settling, the solvent phase was discarded.
- The gas oil raffinate was water-washed back into the original 500 mL bottle with approximately 100 mL of water.
- The water-gas oil raffinate was shaken for 1 hour and then allowed to settle overnight in an oven at 90°C. The bottom water phase was then discarded.
- In an effort to further reduce the gas oil raffinate solvent content, the water wash procedure was repeated.
- Following the second water wash the gas oil-water interface was separately extracted with toluene in an effort to remove all free water. This toluene phase, the remainder of the gas oil and the toluene washings obtained from both separatory funnels and the agitation bottle were quantitatively transferred to a tared 250 mL round bottom flask.
- The toluene-gas oil mixture was refluxed in a Dean and Stark Apparatus (Figure 1) until the last traces of free water were separated via the toluene-water azeotropic distillation.
- The toluene-raffinate mixture was then heated to 170°C and under vacuum to remove the toluene and last traces of water and nitrogen extraction solvent.
- The 250 mL tared flask was reweighed and the final raffinate weight was recorded.
- The percent recovery value was calculated on the basis of the initial 100 g of gas oil feedstock and the final raffinate weight.

2.3 Micro Activity Testing

2.3.1 Micro Activity Testing (MAT) Procedure

FCC processability for all raffinates and gas oil feeds were performed using the MAT apparatus displayed in Figure 2 and were

evaluated against a standard sample of Davison DA-440 regenerated equilibrium catalyst obtained from a Canadian Refinery using Davison Method No. 600 (3).

2.3.2 Derivation of Optimum MAT Parameters

Four catalyst to gas oil ratio (C/O ratio) experiments (Tables 3, 4 and 5) were carried out for each of the three feedstocks and primary Dimethyl Sulfoxide (DMSO) raffinates studied i.e. Canmet 315-524°C, Suncor 360°C+ and Syncrude 360°C+. EM&R provided assistance in obtaining optimum reactor temperature, weight hourly space velocity (WHSV) and C/O ratio for the CANMET gas oil (4).

The optimum MAT C/O ratios for the CANMET, Suncor and Syncrude gas oils and primary DMSO raffinates is given in Tables 6 and 7. As this data was used as the primary source of comparison for the determination of FCC processability, the MAT tests were performed in duplicate and the averaged values were then used as a comparison to MAT results derived from the study case raffinates.

The comparison optimum MAT test parameters used for all Suncor and Syncrude evaluations (Tables 6 and 7) is as follows:

Reactor Temperature	= 489°C
C/O Ratio (g/g)	= 3.6
WHSV	= approx. 11
Catalyst	= Davison DA-440

The comparison optimum MAT test parameters used for all CANMET evaluations (Tables 6 and 7) is as follows:

Reactor Temperature	= 530°C
C/O Ratio (g/g)	= 5.9
WHSV	= approx. 16
Catalyst	= Davison DA-440

2.4 Static extractions with Alternate Aprotic Dipolar Solvents

Previously, gas oil fractions and PDA oils were extracted with N,N-Dimethyl Formamide (DMF) and N-Methyl 2-Pyrrolidone (NMP) with water contents varying from 10-50% in an effort to maximize gas oil nitrogen content removal with respect to solvent water

content. This previous work (see appendum) indicated either DMF or NMP containing 10% water gave maximum nitrogen removal.

In this study, CANMET, Suncor 360°C+, Syncrude IBP-360°C and Syncrude 360°C+ were separately extracted with DMF and NMP containing 10% water (Table 8). MAT evaluation of these raffinates was also carried out (Table 9).

2.5 Optimization of Water Content and Extraction Ratios for Dimethyl Sulfoxide Solvent Systems

CANMET, Suncor 360°C+ and Syncrude 360°C+ were extracted with five DMSO-based extractants containing water at concentrations ranging from 6 to 25 wt% (Tables 10, 11 and 12), The DMSO/H_2O (94:6) solvent system was used to determine the nitrogen-aromatic hydrocarbon partition isotherms for the three gas oil fractions (Tables 10-15). MAT evaluation of DMSO/H_2O (94:6) raffinates while varying the extraction ratio is given in Table 16.

2.6 Multistage Extractions with DMSO/H_2O (94:6)

Using the data obtained from varying the extraction ratio (Section 2.4) and following consultation with the EM&R Scientific Authority, multistage cross-current extractions were carried out on both CANMET (extraction temperature = 20°C) and Syncrude 360°C (extraction temperature = 40°C) gas oil fractions. MAT testing was also performed on the associated raffinates. This data is given in Tables 17-20 and Figures 3 and 4.

2.7 2nd Stage DMSO Extractions with Aqueous 0.2 and 2.0N p-Toluene Sulphonic Acid

Following 1st stage extraction with DMSO/H_2O (94.6) using a 1 to 1 by volume solvent to gas oil extraction ratio, 2nd stage 94% DMSO + 6% aqueous 0.2 and 2.0N p-Toluene Sulphonic Acid extractions were performed. In all, nine such 2nd stage extractions were carried out. This extraction data and associated MAT results is given in Tables 21 and 22.

2.8 Relationship of Temperature - Nitrogen Removal Upon Optimum DMSO Solvent System

Extraction temperatures were separately varied for both CANMET and Syncrude gas oils from 20 to 100°C in 20°C intervals in an agitated

constant temperature bath producing a total of ten temperature related raffinates using a 1 to 1 extraction ratio. Extraction and MAT data is given in Tables 23 and 24.

2.9 Determination of Aprotic Solvents in Raffinates by High Pressure Liquid Chromatography (HPLC)

A HPLC method was developed specifically to determine the levels of DMF, NMP and DMSO remaining in CANMET, Suncor 360°C+ and Syncrude 360°C+ gas oil raffinates. Seventeen samples (Table 25) were analyzed. Details of this method are given in Appendum A.

MAT results of three DMSO adulterated raffinates (10 000 ppm w/w as DMSO) are given in Table 26.

2.10 Characterization of Extracts

Four extracts from a 1st stage CANMET, Suncor and Syncrude DMSO/H_2O (94:6) extractions were selected for compositional characterization.

Initially the four extracts were dissolved in a minimum volume of tetrahydrofuran. The use of the tetrahydrofuran was necessary due to the limited solubility of the extracts. The extracts were then introduced onto a silica gel column. The non-polars were washed off the column with n-hexane and the polars were removed with methylene chloride. Unfortunately the tetrahydrofuran caused both the polar and non-polar fractions to be removed from the column during the initial hexane phase washing, rendering the desired separation of hydrocarbons (non-polars) and nitrogen components (polars) as inadequate.

Elemental analyses (carbon, hydrogen, nitrogen and sulfur) water, basic nitrogen, high resolution mass spectrometry and infra-red analyses were then carried out on the extracts (Table 27). Details of the mass spectrometry elemental and infra-red analyses have been reported in Contract 23SQ.23440-4-9111 (2).

3. RESULTS AND DISCUSSION

Modified extractant compositions and multistaging were carried out in an effort to extend the relationship between gas oil feed FCC processability and nitrogen content (1) for three synthetic feedstocks: CANMET gas oil 315-524°C (Can-F), Syncrude gas oil 360°C+ (Syn-F) and Suncor gas oil 360°C+ (Sun-F). The raffinates produced were submitted for FCC Micro Activity Testing (MAT) for the determination of conversion and product structure yields, and then compared with MAT results obtained from the original feedstocks. Figures 5-7 illustrate observed changes in FCC product yields.

The results of the preliminary tests performed at varying catalyst to oil ratio, C/O, and those of duplication tests are compiled in Tables 3-7. Repeatability data relating to the MAT results is generally satisfactory; however, some determinations do show low percent mass balance values i.e. approx. 95%. Good reproducibility is apparent when CANMET gas oil feed MAT results are compared to the EMR-STA data (4). The average of these duplicate results was used as a standard of comparison for evaluating FCC processability of the various study case raffinates.

Based upon the various C/O ratios in Tables 3, 4 and 5, the following MAT parameters which correspond to moderate cracking and mid point coke yields were selected as the standard conditions for comparison purposes:

- Can-F and related raffinates (T = 530°C; WHSV = 15.8; C/O = 6.0)

- Syn-F and Sun-F and related raffinates: (T = 489°C; WHSV = 11; C/O = 3.6)

The FCC processability of the CANMET, Suncor and Syncrude feedstocks is given in Table 6 and displayed in Figure 5. Can-F showed a different cracking behaviour than either Sun-F and Syn-F, i.e. more coke make, a higher gas yield and lower C_5^+ gasoline product. A comparison of Sun-F and Syn-F showed similar yield structures.

3.1 Extractions with Alternate Aprotic Dipolar Solvents

Tables 8 and 9 show comparison of nitrogen removal capabilities for three solvent systems, i.e. DMF, NMP and DMSO and the improved FCC feedstock properties of the associated raffinates. Maximum denitro- genation and the lowest raffinate recoveries occurredwhen the NMP containing 10% water was used as the extraction solvent. DMF containing 10% of water was slightly less effective than DMSO containing 6% of H_2O.

The extraction efficiency of these solvent systems was assessed by determination of the denitrogenation coefficient, e.g.

$$\text{Denitrogenation Coefficient} = \frac{\% \text{ by wt Nitrogen Removed} \times \% \text{ by wt Raffinate Recovery}}{100}$$

Based upon the denitrogenation coefficient, NMP and DMSO based extractants were equally efficient with Can-F. The latter was superior with Sun-F and Syn-F. DMF appears less effective in all cases as a nitrogen extraction solvent.

All denitrogenations reported led to increases in desirable product yields, i.e. increased C_3 and C_4's and C_5^+ gasoline when compared to MAT data derived from the original gas oil feedstocks. A raffinate to raffinate comparison shows that coke make, gas yield ($\ll C_2$'s) and LCO content did not change significantly. However, denitrogenation did reduce the volume of undesirable decant oil product. NMP based extractant offered the highest reduction in decant oil content. In contrast to DMSO and DMF solvent systems, NMP also increased the ratio of C_5^+ gasoline to conversion for Can-F and Sun-F raffinates. This implies an increase in level of gasoline precursors due to the removal of both nitrogen compounds and polynuclear aromatic hydrocarbons from the original feedstock.

3.2 Optimum Extractions with DMSO Solvent Systems and Multistage Extractions

Tables 10-12 show the relationship between feedstock denitrogenations and raffinate recoveries with varying water content and different extractant to gas oil ratios for DMSO/H_2O solvent system. The denitrogenation coefficient values determined for CANMET raffinates indicate only minor influences for variation in

solvent, water content and extraction ratios relating to optimum nitrogen removal. Optimum denitrogenation or maximum denitrogenation coefficient values appear to have been achieved for solvent as oil extraction ratios of ≮1.2 and solvent water contents of not greater than 6%.

Tables 13, 14 and 15 demonstrate that as the solvent to gas oil extraction ratio is increased more polynuclear aromatic structures are removed from the raffinates derived from CANMET, Suncor and Syncrude sources.

MAT testing performed on the 1/1 and 2/1 raffinates indicated that the solvent ratio did not significantly change the product yields for Syn-F and Sun-F. The 2/1 ratio, however, improved the FCC processability of Can-F, enhancing C_5^+ and C_6^+ gasoline yields (Table 16). These results permitted the choice of DMSO containing 6% water and 1/1 solvent to gas oil extraction ratio as being approximately optimum parameters for multistage extractions.

CANMET and Syncrude multistage extraction potential for maximizing nitrogen removal was evaluated using successive batch extractions designed to simulate a continuous cross-current flow extractor (Table 17). The raffinate nitrogen content was found to decrease with staging. However, 3rd and 4th stage extractions were less effective as the theoretical limit of nitrogen removal was being approached at this point. These equilibrium nitrogen removal limits are displayed graphically in Figures 3 and 4 using Table 18 data.

Based on these equilibrium relationships for nitrogen transfer from the gas oil phase into extractant, complete nitrogen removal does not appear possible with this extractant composition. A possible explanation for this phenomenon could be related to the low affinity of non-extractable nitrogen species, i.e. compounds with sterically hindered heteronitrogen which may resist transfer into the solvent media. The corresponding MATs performed on selected raffinates (Tables 19 and 20) indicated some beneficial yield enhancements (C_3 & C_4's and C_5^+ gasoline) as a result of the successive denitrogenations. However, feedstock improvement beyond 2nd stage extraction is marginal.

Several extractions followed by MATs have been carried out to assess the effect of the extraction temperature on the denitrogenations and product yields. The nitrogen removal was found to

increase slightly with increasing temperature (Table 23) indicating an additional transfer of different nitrogen species than those mobile at ambient temperature. The corresponding FCC yield enhancement of desirable products (Figures 6 and 8, Table 24) also confirmed the positive effect of extractions at elevated temperatures.

3.3 2nd Stage DMSO Extractions with Aqueous 0.2 and 2.0N p-Toluene Sulphonic Acid

By substituting an aqueous p-toluene sulphonic acid solution in place of the 6% water contained in the DMSO, an alternate extraction technique was carried out. These acid extractions were performed on the 1st stage DMSO raffinates, the intent being to increase denitrogenation by enhancing transfer of basic nitrogen species into the acid-DMSO phase. The presence of the acid in DMSO was found to improve considerably denitrogenations (Table 21). Significantly lower nitrogen contents were observed in comparison to 2nd stage raffinates obtained with the H_2O-DMSO system especially for solvent systems containing 2.0N acid concentrations. Consequently improved FCC C_5^+ and C_6^+ gasoline yields were also observed (Table 22, Figures 6, 7 and 8).

3.4 Analyses of Aprotic Solvent Remaining in Gas Oil Raffinates

A high pressure performance liquid chromatography (HPLC) method utilizing a C-18 reverse phase column was developed specifically for the determination of DMF, DMSO and NMP remaining in CANMET, Suncor 360°C+ and Syncrude 360°C+ gas oil raffinates. The aprotic solvent was separated from the gas oil using isocratic elution with 20% acetonitrile and 80% water. The gas oil raffinate was stripped from the column using a backflush technique with 100% tetrahydrofuran followed by 100% acetonitrile. The components of interest were quantitatively determined using external calibration and monitored at 220 nm in the UV. The detection limit of this method is better than 25 ppm by volume (25 ppm v/v DMF ≡ 5 ppm w/w as N; 25 ppm v/v NMP ≡ 4 ppm w/w as N).

Details of the HPLC method are given in Appendum A.

HPLC analyses (Table 25) showed only small quantities (ppm levels) of the extracting aprotic dipolar solvent remained in the water stripped raffinates. NMP appears to be a more difficult solvent to remove from gas oil raffinate than either DMF or DMSO.

3.5 Effect of DMSO Solvent Upon Microactivity Data

In order to evaluate the effect of the residual solvent on raffinate FCC processability, CANMET, Suncor and Syncrude raffinates were doped with 1% DMSO and their micro activity tested. Minor differences in product yields were observed (Table 26) indicating essentially a neutral behaviour from DMSO solvent contamination.

3.6 Compositional Characterization of Extracts

Four extracts were analyzed by elemental and spectroscopic analyses methods to compare their compositional characteristics with those earlier reported (1,2). The extract characteristics are given in Table 27.

As shown in Tables 13-15, the denitrogenations with DMSO solvent systems decreased di, tri, tetra and penta aromatic contents in gas oil raffinates. An increase in the extractant to gas oil ratio results in a lower aromatic raffinate content. Consequently the extracts were not only abundant in nitrogen species, but also contained high levels of aromatics i.e. 2, 3, 4 and 5 condensed ring structures. Basic nitrogen levels appear to constitute only a small portion of the total nitrogen content. A relative comparison of the basic nitrogen content to total nitrogen levels showed CAN-F containing more basic nitrogen than either Suncor and Syncrude extracts. The IR analyses showed high contents of pyrrolic nitrogen species, i.e. carbazoles and benzocarbazoles in all extracts. The extracts also contained a considerable oxygen component, mainly proton donating structures with attached hydroxyl or carboxyl groups. Can-F extracts contained essentially OH compounds, while Syncrude and Suncor oxygenates were dominated by acidic components. Sun-F and Syn-F were also considerably lower in sulfur type structures than the Can-F extracts. In general, the analyses have revealed higher contents of nitrogen in these extracts in comparison to those reported earlier (1,2). Previous results (1,2) indicate that proton donating compounds, in particular components with pyrrolic functionality, are selectively removed from synthetic gas oils. Extraction data contained in this study suggests this type of denitrogenation can be equally effective for both conventional and synthetic gas oil fractions.

4. SUMMARY OF RESULTS

1. Reduction in aprotic solvent water content tends to maximize raffinate total nitrogen content removal while diminishing raffinate recoveries.

2. The most effective solvent-water systems studied in terms of nitrogen component extraction are as follows:
   N,N-Dimethyl Formamide (DMF)/Water (90:10)
   N-Methyl 2-Pyrrolidone (NMP)/Water (90:10)
   Dimethyl Sulfoxide (DMSO)/Water (94:6)

3. 1st stage NMP raffinates have a lower total nitrogen content than either the corresponding DMF or DMSO raffinates.

4. The increased ability of NMP to extract gas oil nitrogen compounds is reflected in improved MAT results, i.e. improved FCC processing capabilities (higher C_5+ gasoline and C_3 and C_4 hydrocarbon yields).

5. Although an increase in the DMSO solvent to gas oil extraction ratio from 1/1 to 2/1 does reduce the raffinate total nitrogen content, the FCC feedstock improvement appears to be only marginally better.

6. An increase of the extraction temperature from 20 to 100°C did not significantly alter the total nitrogen content of either CANMET or Syncrude raffinates, MAT results did show significant improvement for CANMET feedstocks while the Syncrude improvement appeared marginal.

7. 2nd stage DMSO extractions with 6% aqueous 2.0N p-Toluene Sulphonic Acid removed enough basic nitrogen components to significantly reduce the total nitrogen raffinate content of CANMET, Suncor and Syncrude feedstocks. Comparison of MAT results derived from 1st stage DMSO raffinates to 2nd stage DMSO + aqueous p-Toluene Sulphonic Acid did reflect substantial FCC processability improvement. However, comparison of 2nd stage DMSO raffinate MAT results with those obtained from the 2nd stage acid feedstock did not show measurable improvement for Syncrude material, while marginal improvement was noted for CANMET gas oil.

8. Four stage DMSO/H_2O (94:6) cross-current 1 to 1 solvent to gas oil extractions were carried out on CANMET and Syncrude gas oils. The data showed the anticipated continued reduction in raffinate recovery and total nitrogen content. The results further indicated that the 4th stage extraction for CANMET was sufficient to reduce the nitrogen content to the anticipated theoretical equilibrium limit, while the 4th stage Syncrude extraction was not sufficient to reach this calculated nitrogen content minimum level. FCC processability generally improved for each stage of DMSO extraction for both gas oils.

9. Following the development of a HPLC method with a detection limit of less than 25 ppm v/v solvent for the determination of aprotic solvent remaining in gas oil raffinates, seventeen such analyses were carried out. CANMET, Suncor and Syncrude raffinates showed DMF contents from 1-19 ppm v/v (three samples); NMP contents from 1 to 295 ppm v/v (three samples) and DMSO contents ranging from 2 to 61 ppm v/v for 11 samples.

10. MAT tests carried out on DMSO raffinates adulterated with 1% by wt DMSO indicated this aprotic solvent did not significantly alter the anticipated FCC processability.

11. The following conditions appear near optimum for DMSO static extractions with respect to maximizing nitrogen component removal and improved FCC processability:

Solvent Water Content	: 6% by wt
Solvent to Gas Oil Extraction Ratio	: 1 to 1 by volume
Extraction Temperature	: ≅100°C
Number of Extraction Stages	: 1

12. Although in all cases the major nitrogen structures present were pyrrolic types, characterization of four extracts derived from 1st stage CANMET, Suncor and Syncrude DMSO and one 2nd stage Syncrude DMSO extraction showed considerable variation in chemical composition. The total nitrogen compound content varied from 27.9 to 43.5%, while the hydrocarbon component ranged from 45.9 to 67.9%. Sulfur contents varied from 1.13 to 4.04% by wt as S while oxygen ranged from 0.08 to 0.54% by wt as O.

13. Correlation of data obtained from this study and previous work (1,2) indicate selective extractions employing aprotic dipolar solvent systems will partially denitrogenate gas oil fractions derived from both conventional and synthetic sources and improve FCC processability.

14. Due to the solid, waxy nature of the PDA feedstocks and the stability of the water-hydrocarbon emulsions, previous results showed severe raffinate recovery and solvent removal problems during the denitrogenation of these feedstocks. In order to avoid bumping during the rotary-vacuum removal of the wash water used for solvent stripping, the water washed raffinates were initially refluxed in a Dean and Stark apparatus, and free water was removed by a toluene-water azeotropic distillation. This Dean and Stark distillation technique was subsequently used in all extractions carried out in the study and was responsible for the improved raffinate recovery values.

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TABLE 1

GAS OIL FEED DISTILLATIONS

Fraction	% Distilled (by weight)	% Distilled (by volume)
CANMET REACTOR PRODUCT		
IBP -200°C Naphtha	14.94	18.39
200-315°C, Middle Distillate	21.12	22.23
315-524°C, Gas Oil	49.95	48.45
524°C+ Residuum	14.00	10.93
SUNCOR GAS OIL FRACTION		
IBP-360°C	53.55	54.9
360°C+	46.45	45.1
SYNCRUDE GAS OIL FRACTION		
IBP-360°C	28.66	29.64
360°C+	71.34	70.36

TABLE 2

GAS OIL FEED CHARACTERIZATIONS

	CANMET Gas Oil (315-524°C)	Suncor Gas Oil (360°C+)	Syncrude Gas Oil (IBP-360°C)	Syncrude Gas Oil (360°C+)
Basic Nitrogen, ppm w/w as N	1960	293	90	230
Total Nitrogen, ppm w/w as N	4438	1336	280	1473
Total Sulfur, % by Wt. as S	2.57	0.66	0.14	0.44
Density, @ 15°C	0.9698	0.9422	0.9082	0.9496
Gas Chromatographic Distillation, °C				
IBP	196	334	182	333
5%	325	354	224	357
10%	338	364	246	369
15%	349	372	259	377
20%	359	378	270	385
25%	369	384	278	392
30%	377	389	286	399
35%	385	394	294	406
40%	393	399	301	414
45%	401	405	308	421
50%	408	410	313	428
55%	416	415	319	435
60%	424	421	324	443
65%	432	426	328	451
70%	440	433	333	460
75%	450	440	338	470
80%	460	447	343	481
85%	471	457	348	493
90%	484	469	353	509
90%	502	486	360	535
FBP	544	532	412	615

TABLE 3

MICROACTIVITY TEST RESULTS
FCC CATALYST TO GAS OIL RATIO DATA
CANMET GAS OIL AND DMSO RAFFINATE

Run No.	1	2	3	4	5	6	7	8
				wt %				
Reactor Temp. °C	530	530	530	530	530	530	530	530
WHSV	22.2	15.8	10.9	9.3	23.6	14.8	11.9	8.8
C/O	4.0	5.7	8.3	9.6	3.8	6.1	7.6	10.2
Reactor Effluent Gas								
Hydrogen	0.10	0.18	0.22	0.23	0.10	0.16	0.14	0.22
Methane	0.65	1.12	1.79	1.93	0.67	1.34	1.35	2.01
C_2 Hydrocarbons	1.05	1.72	2.31	2.61	1.15	2.08	2.02	2.86
C_3 Hydrocarbons	3.49	5.28	6.88	7.09	4.68	6.84	6.91	8.17
C_4 Hydrocarbons	4.15	6.46	8.97	8.74	6.38	9.00	9.53	10.9
C_5 Hydrocarbons	3.50	4.86	7.29	7.00	4.93	6.52	7.20	8.10
C_6 Hydrocarbons	1.24	1.34	2.31	2.01	1.47	1.59	1.88	2.13
C_6^+ Hydrocarbons	1.93	2.11	3.60	2.53	2.26	1.97	2.80	2.61
Product Cuts								
C_2 & lighter	2.13	3.22	4.62	5.04	2.43	4.04	3.86	5.87
C_3 & C_4	7.64	11.8	15.9	15.8	11.1	15.8	16.4	19.1
C_5^+ Gasoline (220 EP)	26.5	32.6	35.2	34.3	32.2	35.3	39.2	36.6
C_6^+ Gasoline (220 EP)	23.0	27.8	27.8	27.3	27.3	28.8	31.9	28.5
LCO (220-344°C)	23.9	22.4	17.7	18.9	22.1	20.2	18.4	16.3
Decant (344°C+)	35.3	22.8	13.7	13.1	28.0	17.2	12.4	9.1
Coke Make	4.5	7.2	13.0	12.8	4.2	7.4	9.8	13.2
Coke on Catalyst	1.1	1.2	1.6	1.3	1.1	1.2	1.3	1.3
Conversion	40.8	54.8	68.6	68.0	49.9	62.6	69.2	74.7
Material Balance	100.8	98.6	100.7	98.3	99.0	96.7	99.4	97.0

1 - CANMET Gas Oil, C/O = 4.0
2 - CANMET Gas Oil, C/O = 5.7
3 - CANMET Gas Oil, C/O = 8.3
4 - CANMET Gas Oil, C/O = 9.6
5 - CANMET DMSO/H_2O (94:6), C/O = 3.8
6 - CANMET DMSO/H_2O (94:6), C/O = 6.1
7 - CANMET DMSO/H_2O (94:6), C/O = 7.6
8 - CANMET DMSO/H_2O (94:6), C/O = 10.2

TABLE 4

MICROACTIVITY TEST RESULTS
FCC CATALYST TO GAS OIL RATIO DATA
SUNCOR GAS OIL AND DMSO RAFFINATE

wt%

Run No.	1	2	3	4	5	6	7	8
Reactor Temp. °C	489	489	489	489	489	489	489	489
WHSV	21.5	11.0	6.3	4.8	20.6	10.2	6.6	4.7
C/O	1.9	3.6	6.4	8.3	1.9	3.9	6.0	8.4
Reactor Effluent Gas								
Hydrogen	0.04	0.07	0.09	0.11	0.03	0.06	0.09	0.05
Methane	0.25	0.60	0.88	1.07	0.24	0.66	0.84	0.74
C_2 Hydrocarbons	0.54	1.14	1.74	1.84	0.57	1.30	1.66	1.38
C_3 Hydrocarbons	3.13	5.52	6.76	6.73	3.46	6.02	6.15	5.92
C_4 Hydrocarbons	5.10	8.75	11.0	11.1	5.57	9.69	10.6	10.4
C_5 Hydrocarbons	3.25	5.64	7.25	7.92	2.82	5.95	6.91	8.71
C_6 Hydrocarbons	0.66	1.26	1.13	1.64	0.45	1.14	1.26	2.55
C_6^+ Hydrocarbons	0.88	1.93	1.51	2.09	0.65	1.70	1.68	4.00
Product Cuts								
C_2 & lighter	0.90	1.86	2.84	3.15	0.93	2.13	2.78	2.21
C_3 & C_4	8.23	14.2	17.8	17.9	9.03	15.7	16.8	16.3
C_5^+ Gasoline (220 EP)	33.4	45.1	42.4	45.3	46.8	49.1	48.6	52.8
C_6^+ Gasoline (220 EP)	30.1	39.5	35.2	37.4	44.0	43.2	41.7	44.0
LCO (220-344°C)	20.4	21.1	19.7	17.0	22.7	19.3	17.6	15.8
Decant (344°C+)	35.6	14.1	10.7	8.0	18.6	9.8	7.73	5.1
Coke Make	1.6	3.5	6.6	8.7	2.0	3.9	6.5	7.8
Coke on Catalyst	0.8	1.0	1.0	1.0	1.0	1.0	1.1	0.9
Conversion	44.0	64.8	69.6	75.1	58.7	70.8	74.7	79.1
Material Balance	98.2	97.8	95.4	97.7	98.6	96.5	98.7	104.0

1 - Suncor Gas Oil, C/O = 1.9
2 - Suncor Gas Oil, C/O = 3.6
3 - Suncor Gas Oil, C/O = 6.3
4 - Suncor Gas Oil, C/O = 8.3
5 - Suncor + DMSO/H_2O (94:6), C/O = 1.9
6 - Suncor + DMSO/H_2O (94:6), C/O = 3.9
7 - Suncor + DMSO/H_2O (94:6), C/O = 6.0
8 - Suncor + DMSO/H_2O (94:6), C/O = 8.4

TABLE 5

MICROACTIVITY TEST RESULTS
FCC CATALYST TO GAS OIL RATIO DATA
SYNCRUDE GAS OIL AND DMSO RAFFINATE

wt%

Run No.	1	2	3	4	5*	6	7	8
Reactor Temp. °C	489	490	489	489	489	489	489	489
WHSV	19.8	10.7	6.9	4.7	20.8	11.2	7.1	4.9
C/O	2.0	3.8	5.8	8.4	1.9	3.6	5.7	8.2
Reactor Effluent Gas								
Hydrogen	0.04	0.07	0.10	0.12	0.02	0.06	0.09	0.12
Methane	0.26	0.53	0.89	1.13	0.31	0.66	0.90	1.14
C_2 Hydrocarbons	0.58	0.98	1.62	1.93	0.64	1.22	1.66	1.93
C_3 Hydrocarbons	3.09	4.84	5.88	6.66	3.07	5.34	6.03	6.94
C_4 Hydrocarbons	5.16	7.38	9.48	11.0	4.72	8.66	9.82	11.9
C_5 Hydrocarbons	3.37	4.98	5.84	8.35	2.32	5.36	5.85	9.96
C_6 Hydrocarbons	0.89	0.98	1.12	1.88	0.48	1.02	1.08	2.72
C_6^+ Hydrocarbons	1.48	1.76	1.62	2.35	0.78	1.44	1.63	3.44
Product Cuts								
C_2 & lighter	0.90	1.62	2.65	3.19	0.99	1.96	2.70	3.25
C_3 & C_4	8.25	12.8	15.4	17.7	7.79	14.0	15.9	18.8
C_5^+ Gasoline (220 EP)	41.0	47.4	46.4	45.6	37.1	49.0	46.5	46.6
C_6^+ Gasoline (220 EP)	37.6	42.4	40.6	37.3	34.8	44.0	40.6	36.7
LCO (220-344°C)	23.3	21.2	19.1	17.1	20.3	20.2	18.6	16.2
Decant (344°C+)	24.6	12.3	9.3	7.3	31.6	10.6	9.3	6.3
Coke Make	2.0	4.0	7.2	9.1	2.1	4.3	7.1	8.9
Coke on Catalyst	1.0	1.1	1.2	1.1	1.1	1.2	1.2	1.1
Conversion	52.1	66.0	71.7	75.6	48.0	69.2	72.1	77.6
Material Balance	100.5	100.1	98.3	97.8	99.2	100.2	98.7	97.9

1 - Syncrude Gas Oil, C/O = 2.0
2 - Syncrude Gas Oil, C/O = 3.8
3 - Syncrude Gas Oil, C/O = 5.8
4 - Syncrude Gas Oil, C/O = 8.4
5 - Syncrude DMSO/H_2O (94:6), C/O = 1.9
6 - Syncrude DMSO/H_2O (94:6), C/O = 3.6
7 - Syncrude DMSO/H_2O (94:6), C/O = 5.7
8 - Syncrude DMSO/H_2O (94:6), C/O = 8.2

TABLE 6

MICROACTIVITY TEST RESULTS
GAS OIL FEED DUPLICATION DATA
wt%

Run No.	1	2	Can-F	3	4	Sun-F	5	6	Syn-F
Reactor Temp. °C	529	530	530	489	489	489	489	490	490
WHSV	15.8	15.7	15.8	11.0	10.9	11.0	10.9	10.5	10.7
C/O	5.7	5.7	5.7	3.6	3.7	3.6	3.7	3.8	3.8
Reactor Effluent Gas									
Hydrogen	0.22	0.15	0.18	0.09	0.05	0.07	0.08	0.06	0.07
Methane	1.29	0.95	1.12	0.76	0.43	0.60	0.61	0.45	0.53
C_2 Hydrocarbons	1.93	1.50	1.72	1.39	0.90	1.14	1.16	0.90	0.98
C_3 Hydrocarbons	5.60	4.97	5.28	6.28	4.76	5.52	5.05	4.63	4.84
C_4 Hydrocarbons	6.57	6.34	6.46	9.64	7.86	8.75	8.27	7.68	7.38
C_5 Hydrocarbons	4.72	5.00	4.86	5.82	5.45	5.64	5.28	4.68	4.98
C_6 Hydrocarbons	1.19	1.48	1.34	1.15	1.36	1.26	1.20	0.77	0.98
C_6^+ Hydrocarbons	1.84	2.38	2.11	1.81	2.05	1.93	1.84	1.67	1.76
Product Cuts									
C_2 & lighter	3.66	2.78	3.22	2.28	1.44	1.86	1.84	1.41	1.62
C_3 & C_4	12.2	11.3	11.8	15.9	12.6	14.2	13.3	12.3	12.8
C_5^+ Gasoline (220 EP	33.2	32.1	32.6	43.9	46.3	45.1	48.2	46.7	47.4
C_6^+ Gasoline (220 EP	28.5	27.1	27.8	38.1	40.9	39.5	42.9	42.0	42.4
LCO (220-344°C)	22.5	22.3	22.4	20.4	21.8	21.1	20.5	21.9	21.2
Decant (344°C+)	21.1	24.6	22.8	13.7	14.5	14.1	11.7	13.9	12.3
Coke Make	7.4	7.0	7.2	3.7	3.3	3.5	4.3	3.8	4.0
Coke on Catalyst	1.3	1.2	1.2	1.0	1.1	1.0	1.2	1.0	1.1
Conversion	56.5	53.1	54.8	65.8	63.7	64.8	67.7	64.2	66.0
Material Balance	98.1	99.2	98.6	95.5	100.1	97.8	99.8	100.4	100.1

1 - CANMET Gas Oil Feed Run #1
2 - CANMET Gas Oil Feed Run #2
Can-F - Average (2 runs) Canmet Feed
3 - Suncor Gas Oil Feed Run #1
4 - Suncor Gas Oil Feed Run #2
Sun-F - Average (2 runs) Suncor Feed
5 - Syncrude Gas Oil Feed Run #1
6 - Syncrude Gas Oil Feed Run #2
Syn-F - Average (2 runs) Syncrude Feed

TABLE 7

MICROACTIVITY TEST RESULTS

DMSO/H_2O (94:6) RAFFINATE DUPLICATION DATA

wt%

Run No.	1	2	Can-R	3	4	Sun-R	5	6	Syn-R
Reactor Temp. °C	531	530	530	489	489	489	489	489	489
WHSV	14.7	14.8	14.8	9.9	10.4	10.2	11.4	10.9	11.2
C/O	6.1	6.1	6.1	4.0	3.8	3.9	3.5	3.7	3.6
Reactor Effluent Gas									
Hydrogen	0.16	0.17	0.16	0.07	0.05	0.06	0.05	0.07	0.06
Methane	1.35	1.34	1.34	0.83	0.49	0.66	0.63	0.68	0.66
C_2 Hydrocarbons	2.10	2.07	2.08	1.48	1.13	1.30	1.23	1.22	1.22
C_3 Hydrocarbons	7.24	6.43	6.84	6.34	5.69	6.02	5.50	5.18	5.34
C_4 Hydrocarbons	9.57	8.44	9.00	10.2	9.18	9.69	9.02	8.30	8.66
C_5 Hydrocarbons	6.97	6.06	6.52	6.08	5.81	5.95	5.94	4.77	5.36
C_6 Hydrocarbons	1.87	1.31	1.59	1.17	1.10	1.14	1.14	0.91	1.02
C_6^+ Hydrocarbons	2.24	1.70	1.97	1.53	1.87	1.70	1.74	1.13	1.44
Product Cuts									
C_2 & lighter	3.87	4.21	4.04	2.46	1.80	2.13	1.94	1.97	1.96
C_3 & C_4	16.8	14.9	15.8	16.5	14.9	15.7	14.5	13.5	14.0
C_5^+ Gasoline (220 EP)	37.6	33.0	35.3	49.1	49.1	49.1	49.1	48.9	49.0
C_6^+ Gasoline (220 EP)	30.6	26.9	28.8	43.1	43.3	43.2	43.7	44.2	44.0
LCO (220-344°C)	20.0	20.5	20.2	18.5	20.1	19.4	20.3	20.2	20.2
Decant (344°C+)	14.2	20.2	17.2	8.97	10.7	9.8	10.1	11.0	10.6
Coke Make	7.5	7.3	7.4	4.42	3.4	3.9	4.1	4.5	4.3
Coke on Catalyst	1.2	1.2	1.2	1.1	0.9	1.0	1.1	1.2	1.2
Conversion	65.8	59.3	62.6	72.5	69.2	70.8	69.6	68.8	69.2
Material Balance	98.0	95.4	96.7	97.7	95.3	96.5	99.0	100.2	99.6

1 - CANMET Raffinate Run #1

2 - CANMET Raffinate Run #2

Can-R - Average (2 runs) Canmet Raffinate

3 - Suncor Raffinate Run #1

4 - Suncor Raffinate Run #2

Sun-r - Average (2 runs) Suncor Raffinate

5 - Syncrude Raffinate Run #1

6 - Syncrude Raffinate Run #2

Syn-R - Average (2 runs) Syncrude Raffinate

TABLE 8

COMPARISON OF SOLVENT SYSTEMS NITROGEN REMOVAL CAPABILITIES

	CANMET (315-524°C)		Suncor (360°C+)		Syncrude (IBP-360°C)		Syncrude (360°C+)	
	Gas Oil Feed	Raffinate	Gas Oil Feed	Raffinate	Gas Oil Feed	Raffinate	Gas Oil Feed	Raffinate
N-Methyl 2-Pyrrolidone + 10% Water								
Total Nitrogen (ppm w/w as N)	4438	1884	1336	577	280	98	1473	786
Raffinate Recovery (% wt)	-	82.7	-	91.7	-	92.6	-	94.0
Denitrogenation Coefficient		47.6		52.1		-		43.8
N,N-Dimethyl Formamide + 10% Water								
Total Nitrogen (ppm w/w as N)	4438	2414	1336	798	280	105	1473	877
Raffinate Recovery (% wt)	-	82.6	-	93.3	-	91.7	-	93.5
Denitrogenation Coefficient		37.7		37.6		-		37.8
Dimethyl Sulfoxide + 10% Water								
Total Nitrogen (ppm w/w as N)	4438	2800	1336	759	280	N.A.	1473	944
Raffinate Recovery (% wt)	-	89.0	-	97.2	-	N.A.	-	97.9
Denitrogenation Coefficient		32.8		42.0				35.2
Dimethyl Sulfoxide + 6% Water								
Total Nitrogen (ppm w/w as N)	4438	2464	1336	650	280	N.A.	1473	854
Raffinate Recovery (% wt)	-	86.8	-	97.2	-	N.A.	-	97.0
Denitrogenation Coefficient		38.6		49.9				40.8

N.A. = Not available

TABLE 9

MICROACTIVITY TEST RESULTS
COMPARISON OF SOLVENT SYSTEMS NITROGEN REMOVAL CAPABILITIES

wt%

Run No.	Can-F	2	3	Can-R	Sun-F	4	5	Sun-R	Syn-F	6	7	Syn-R
Reactor Temp. °C	530	529	529	530	489	489	489	489	490	489	489	489
WHSV	15.8	16.5	16.6	14.8	11.0	10.8	10.8	10.2	10.7	11.3	11.1	11.2
Catalyst/Oil Ratio	5.7	5.5	5.4	6.1	3.6	3.7	3.7	3.9	3.8	3.5	3.6	3.6
Reactor Effluent Gas												
Hydrogen	0.18	0.23	0.11	0.16	0.07	0.04	0.03	0.06	0.07	0.06	0.09	0.06
Methane	1.12	1.83	1.17	1.34	0.60	0.67	0.53	0.66	0.53	0.57	0.85	0.66
C_2 Hydrocarbons	1.72	2.43	2.07	2.08	1.14	1.36	1.20	1.30	0.98	1.19	1.45	1.22
C_3 Hydrocarbons	5.28	7.28	7.54	6.84	5.52	5.89	6.30	6.02	4.84	5.42	6.07	5.34
C_4 Hydrocarbons	6.46	9.02	10.7	9.00	8.75	9.40	10.7	9.69	7.38	9.01	9.53	8.66
C_5 Hydrocarbons	4.86	6.69	6.95	6.52	5.64	6.14	6.94	5.95	4.98	5.43	5.41	5.36
C_6 Hydrocarbons	1.34	1.88	1.41	1.59	1.26	1.34	1.43	1.14	0.98	1.03	1.03	1.02
C_6^+ Hydrocarbons	2.11	2.81	1.70	1.97	1.93	2.17	2.35	1.70	1.76	1.25	1.59	1.44
Product Cuts												
C_2 & lighter	3.22	4.78	3.99	4.04	1.86	2.13	1.86	2.13	1.62	1.86	2.42	1.96
C_3 & C_4	11.8	16.3	18.2	15.8	14.2	15.3	16.7	15.7	12.8	14.4	15.6	14.0
C_5^+ Gasoline (220 EP)	32.6	38.1	42.8	35.3	45.1	48.4	51.2	49.1	47.4	49.4	51.5	49.0
C_6^+ Gasoline (220 EP)	27.8	31.4	35.9	28.8	39.5	42.2	44.3	43.2	42.4	44.0	46.1	44.0
LCO (220-344°C)	22.4	18.0	18.7	20.2	21.2	19.9	18.8	19.3	21.2	20.2	18.0	20.2
Decant (344°C+)	22.8	14.4	10.4	17.2	14.1	10.1	8.1	9.8	12.3	10.5	8.4	10.6
Coke Make	7.2	8.4	5.9	7.4	3.5	4.3	3.1	3.9	4.0	3.7	4.1	4.3
Coke on Catalyst	1.2	1.5	1.1	1.2	1.0	1.1	0.8	1.0	1.1	1.0	1.1	1.2
Conversion	54.8	67.6	70.9	62.6	64.8	70.0	73.2	70.8	66.0	69.3	73.6	69.2
Material Balance	98.6	95.9	96.7	96.7	97.8	98.0	95.9	96.5	100.1	96.4	98.7	99.6

Can-F - CANMET Gas Oil Feed (Averaged
2 - CANMET DMF/H_2O (90:10) Raffinate
3 - CANMET NMP/H_2O (90:10) Raffinate
Can-R - CANMET DMSO/H_2O (94:6) Raffinate (Averaged)
Sun-F - Suncor Gas Oil Feed (Averaged)
4 - Suncor DMF/H_2O (90:10) Raffinate
5 - Suncor NMP/H_2O (90:10) Raffinate
Sun-R - Suncor DMSO/H_2O (94:6) Raffinate (Averaged)
Syn-F - Syncrude Gas Oil Feed (Averaged)
6 - Syncrude DMF/H_2O (90:10) Raffinate
7 - Syncrude NMP/H_2O (90:10) Raffinate
Syn-R - Syncrude DMSO/H_2O (94:6) Raffinate (Averaged)

TABLE 10

CANMET GAS OIL - DIMETHYL SULPHOXIDE EXTRACTIONS

DMSO Water Content * (wt%)	Total Nitrogen (ppm w/w as N)	Recovery (wt%)	Denitrogenation Coeff.
Feed	4438	-	-
6	2464	86.8	38.6
10	2800	89.0	32.9
15	3265	92.4	24.4
20	3593	95.4	18.2
25	3818	96.6	13.5

* A 1/1 by volume extraction ratio of DMSO + water to CANMET gas oil was used to determine the effect of water on nitrogen content removal.

DMSO/H_2O (94:6) CANMET Gas Oil Extraction Ratio (by vol.)	Total Nitrogen (ppm w/w as N)	Recovery (wt%)	Denitrogenation Coeff.
Feed	4438	-	-
0.25/1	3175	90.0	25.6
0.50/1	2836	88.5	31.9
0.75/1	2470	88.0	39.0
1/1	2464	86.8	38.6
1.5/1	2059	85.6	45.9
2.0/1	1954	85.1	47.6

TABLE 11

SUNCOR GAS OIL - DIMETHYL SULPHOXIDE EXTRACTIONS

DMSO Water Content * (wt%)	Total Nitrogen (ppm w/w as N)	Recovery (wt%)	Denitrogenation Coeff.
Feed	1336	-	-
6	650	97.2	49.9
10	759	97.2	42.0
15	865	97.5	34.4
20	1030	96.9	22.2
25	1048	98.5	21.2

* A 1/1 by volume extraction ratio of DMSO + water to Suncor gas oil was used to determine the effect of water on nitrogen content removal.

DMSO/H_2O (94:6) Suncor Gas Oil Extraction Ratio (by vol.)	Total Nitrogen (ppm w/w as N)	Recovery (wt%)	Denitrogenation Coeff.
Feed	1336	-	-
0.25/1	1049	99.7	21.4
0.50/1	862	97.5	34.6
0.75/1	782	97.9	40.6
1/1	650	97.2	49.9
1.5/1	607	97.0	52.9
2.0/1	594	95.5	53.0

TABLE 12

SYNCRUDE GAS OIL - DIMETHYL SULPHOXIDE EXTRACTIONS

DMSO Water Content * (wt%)	Total Nitrogen (ppm w/w as N)	Recovery (wt%)	Denitrogenation Coeff.
Feed	1473	-	-
6	854	97.0	40.8
10	944	97.9	35.2
15	1120	98.2	23.5
20	1220	97.6	16.8
25	1400	97.5	4.8

* A 1/1 by volume extraction ratio of DMSO + water to Syncrude gas oil was used to determine the effect of water on nitrogen content removal.

$DMSO/H_2O$ (94:6) Syncrude Gas Oil Extraction Ratio (by vol.)	Total Nitrogen (ppm w/w as N)	Recovery (wt%)	Denitrogenation Coeff.
Feed	1473	-	-
0.25/1	1221	99.2	17.0
0.50/1	1054	96.4	27.4
0.75/1	891	98.1	38.8
1/1	854	97.0	40.8
1.5/1	735	98.2	49.2
2.0/1	701	93.2	48.8

TABLE 13

CANMET GAS OIL - DIMETHYL SULPHOXIDE EXTRACTIONS

M.S. ANALYSES % BY WT

DMSO/H_2O (94:6)-Gas Oil Extraction Ratio (by vol.)	Mono-aromatics	Di-aromatics	Tri-aromatics	Tetra-aromatics	Penta-aromatics	Poly-aromatics	Total Aromatics
Feed	14.1	16.2	9.3	5.0	3.5	0.1	48.2
0.25/1	15.4	16.3	10.2	4.4	2.1	0.2	48.4
0.50/1	15.5	15.7	8.8	2.9	0.5*	0.0	43.4
0.75/1	15.6	14.5	9.8	4.7	3.2	0.0	47.8
1/1	15.5	14.0	10.0	2.4	2.8	0.2	44.9
1.5/1	15.8	14.9	8.8	3.1	2.6	0.0	45.2
2.0/1	17.2	14.5	8.5	3.0	1.7	0.0	44.9

* Result is suspect.

TABLE 14

SUNCOR GAS OIL - DIMETHYL SULPHOXIDE EXTRACTIONS

M.S. ANALYSES % BY WT

DMSO/H_2O (94:6)-Gas Oil Extraction Ratio (by vol.)	Mono-aromatics	Di-aromatics	Tri-aromatics	Tetra-aromatics	Penta-aromatics	Poly-aromatics	Total Aromatics
Feed	18.3	17.4	9.8	4.3	1.9	0.0	51.7
0.25/1	17.0	15.3	9.3	3.9	1.4	0.0	46.6
0.50/1	20.8	16.6	8.6	3.0	1.2	0.0	50.2
0.75/1	22.5	16.4	9.6	3.1	1.4	0.0	53.0
1/1	20.5	16.3	8.8	2.2	0.5	0.0	48.3
1.5/1	22.8	17.0	9.2	3.0	0.3	0.0	52.3
2.0/1	22.2	17.3	8.7	2.4	0.0	0.0	50.6

TABLE 15

SYNCRUDE GAS OIL - DIMETHYL SULPHOXIDE EXTRACTIONS

M.S. ANALYSES % BY WT

DMSO/H_2O (94:6)-Gas Oil Extraction Ratio (by vol.)	Mono-aromatics	Di-aromatics	Tri-aromatics	Tetra-aromatics	Penta-aromatics	Poly-aromatics	Total Aromatics
Feed	23.4	19.1	9.2	4.0	1.0	0.0	56.7
0.25/1	24.4	18.7	9.4	3.5	1.0	0.0	57.0
0.50/1	24.7	18.3	9.1	2.8	0.7	0.0	55.6
0.75/1	24.9	18.2	8.8	2.6	0.3	0.0	54.8
1/1	24.8	17.7	7.6	3.0	1.2	0.0	54.3
1.5/1	24.4	17.5	7.5	2.4	0.7	0.0	52.5
2.0/1	25.8	17.9	7.4	1.9	0.4	0.0	53.4

TABLE 16

MICROACTIVITY TEST RESULTS

VARIATION IN DMSO/H_2O (94:6) SOLVENT TO GAS OIL EXTRACTION RATIO

wt%

Run No.	Can-F	1	2	Sun-F	3	4	Syn-F	5	6
Reactor Temp. °C	530	530	529	489	489	489	490	489	489
WHSV	15.8	14.8	14.7	11.0	10.2	11.1	10.9	11.2	12.0
C/O	5.7	6.1	6.1	3.6	3.9	3.9	3.8	3.6	3.3
Reactor Effluent Gas									
Hydrogen	0.18	0.16	0.23	0.07	0.06	0.07	0.07	0.06	0.05
Methane	1.12	1.34	1.73	0.60	0.66	0.69	0.53	0.66	0.47
C_2 Hydrocarbons	1.72	2.08	2.54	1.14	1.30	1.41	0.98	1.22	1.04
C_3 Hydrocarbons	5.28	6.84	7.74	5.52	6.02	6.45	4.84	5.34	4.84
C_4 Hydrocarbons	6.46	9.00	9.90	8.75	9.69	10.3	7.38	8.66	8.04
C_5 Hydrocarbons	4.86	6.52	6.64	5.64	5.95	6.10	4.98	5.36	4.45
C_6 Hydrocarbons	1.34	1.59	1.38	1.26	1.14	1.03	0.98	1.02	0.73
C_6+ Hydrocarbons	2.11	1.97	1.72	1.93	1.70	1.34	1.76	1.44	0.99
Product Cuts									
C_2 & lighter	3.22	4.04	4.79	1.86	2.13	2.27	1.62	1.96	1.61
C_3 & C_4	11.8	15.8	17.7	14.2	15.7	16.8	12.8	14.0	12.9
C_5+ Gasoline (220 EP)	32.6	35.3	40.2	45.1	49.1	50.1	47.4	49.0	50.8
C_6+ Gasoline (220 EP)	27.8	28.8	33.6	39.5	43.2	44.0	42.4	44.0	46.4
LCO (220-344°C)	22.4	20.2	17.8	21.1	19.3	18.4	21.2	20.2	20.8
Decant (344°C+)	22.8	17.2	12.0	14.1	9.8	9.0	12.3	10.6	10.4
Coke Make	7.2	7.4	7.6	3.5	3.9	3.5	4.0	4.3	3.5
Coke on Catalyst	1.2	1.2	1.2	1.0	1.0	1.0	1.1	1.2	1.0
Conversion	54.8	62.6	70.3	64.8	70.8	72.6	66.0	69.2	68.8
Material Balance	98.6	96.7	97.3	97.8	96.5	95.1	100.1	100.2	98.4

1 - CANMET Raffinate 1/1 Extraction Ratio

2 - CANMET Raffinate 2/1 Extraction Ratio

3 - Suncor Raffinate 1/1 Extraction Ratio

4 - Suncor Raffinate 2/1 Extraction Ratio

5 - Syncrude Raffinate 1/1 Extraction Ratio

6 - Syncrude Raffinate 2/1 Extraction Ratio

TABLE 17

CANMET GAS OIL

CROSS CURRENT DMSO/H_2O (94:6) EXTRACTIONS

(1 to 1 by volume extraction ratio)

Temperature = 20°C

Gas Oil Fraction	Total Nitrogen ppm w/w as N	Recovery (wt%)
CANMET Gas Oil Feed	4438	-
CANMET 1st Stage Raffinate	2468	86.8
CANMET 2nd Stage Raffinate	1903	83.0
CANMET 3rd Stage Raffinate	1606	82.8
CANMET 4th Stage Raffinate	1434	80.8

SYNCRUDE GAS OIL

CROSS CURRENT DMSO/H_2O (94:6) EXTRACTIONS

(1 to 1 by volume extraction ratio)

Temperature = 40°C

Gas Oil Fraction	Total Nitrogen ppm w/w as N	Recovery (wt%)
Syncrude Gas Oil Feed	1473	-
Syncrude 1st Stage Raffinate	691	97.0
Syncrude 2nd Stage Raffinate	565	94.1
Syncrude 3rd Stage Raffinate	490	92.6
Syncrude 4th Stage Raffinate	421	90.2

TABLE 18

GAS OIL - DMSO/H_2O (94:6) - NITROGEN CONTENT
EQUILIBRIUM DATA

Solvent to Oil Extraction Ratio (by volume)	Gas Oil Total Nitrogen Content (mg/kg as N)	Solvent Total Nitrogen Content (mg/kg as N)
CANMET GAS OIL		
Feed	4438	0
0.25/1	3175	3437
0.50/1	2836	2437
0.75/1	2470	2104
1/1	2464	1619
1.5/1	2059	1341
2.0/1	1954	1069
SYNCRUDE GAS OIL		
Feed	1473	0
0.25/1	1221	921
0.50/1	1054	767
0.75/1	891	710
1/1	854	532
1.5/1	735	448
2.0/1	701	353

DMSO/H_2O (94:6), Density @ 20°C = 1.093

TABLE 19

MICROACTIVITY TEST RESULTS
CANMET CROSS CURRENT DMSO/H2O (94:6), MULTISTAGE EXTRACTIONS
wt%

Run No.	Can-F	Can-R	1	2	3
Reactor Temp. °C	530	530	530	530	530
WHSV	15.8	14.8	15.9	15.2	14.9
C/O	5.7	6.1	5.7	5.9	6.0
Reactor Effluent Gas					
Hydrogen	0.18	0.16	0.11	0.14	0.13
Methane	1.12	1.34	1.12	1.64	1.60
C_2 Hydrocarbons	1.72	2.08	1.75	2.15	2.01
C_3 Hydrocarbons	5.28	6.84	6.95	7.99	7.76
C_4 Hydrocarbons	6.46	9.00	10.4	11.3	11.5
C_5 Hydrocarbons	4.86	6.52	7.56	8.34	7.7
C_6 Hydrocarbons	1.34	1.59	1.85	2.16	2.34
C_6+ Hydrocarbons	2.11	1.97	2.27	3.15	2.34
Product Cuts					
C_2 & lighter	3.22	4.04	3.33	4.37	4.48
C_3 & C_4	11.8	15.8	17.3	19.3	19.3
C_5+ Gasoline (220 EP)	32.6	35.3	40.8	40.9	41.9
C_6+ Gasoline (220 EP)	27.8	28.8	33.2	32.6	34.2
LCO (220-344°C)	22.4	20.2	19.2	17.2	17.6
Decant (344°C+)	22.8	17.2	13.1	10.2	10.7
Coke Make	7.2	7.4	6.2	8.0	6.1
Coke on Catalyst	1.2	1.2	1.1	1.3	1.0
Conversion	54.8	62.6	67.7	72.6	71.3
Material Balance	98.6	96.7	95.3	95.5	94.1

1 - CANMET 2nd stage Raffinate
2 - CANMET 3rd stage Raffinate
3 - CANMET 4th stage Raffinate

TABLE 20

MICROACTIVITY TEST RESULTS
SYNCRUDE CROSS CURRENT DMSO/H_2O (94:6) MULTISTAGE EXTRACTIONS

Run No.	Syn-F	wt% Syn-R	1	2*	3
Reactor Temp. °C	490	489	489	489	489
WHSV	10.7	11.2	11.1	11.4	11.4
C/O	3.8	3.6	3.6	3.5	3.9
Reactor Effluent Gas					
Hydrogen	0.07	0.06	0.05	0.06	0.04
Methane	0.53	0.66	0.60	0.41	0.53
C_2 Hydrocarbons	0.98	1.22	1.30	0.98	1.18
C_3 Hydrocarbons	4.84	5.34	5.83	4.66	5.67
C_4 Hydrocarbons	7.38	8.66	9.63	7.39	9.65
C_5 Hydrocarbons	4.98	5.36	5.83	3.86	6.03
C_6 Hydrocarbons	0.98	1.02	1.29	0.69	1.18
C_6^+ Hydrocarbons	1.76	1.44	1.90	1.36	1.49
Product Cuts					
C_2 & lighter	1.62	1.96	2.00	1.47	1.83
C_3 & C_4	12.8	14.0	15.5	12.1	15.3
C_5^+ Gasoline (220 EP)	47.4	49.0	50.6	51.4	52.4
C_6^+ Gasoline (220 EP)	42.4	44.0	44.8	47.5	46.4
LCO (220-344°C)	21.2	20.2	18.9	20.7	18.8
Decant (344°C+)	12.3	10.6	8.8	10.9	8.3
Coke Make	4.0	4.3	4.2	3.6	3.4
Coke on Catalyst	1.1	1.2	1.1	1.0	1.0
Conversion	66.0	69.2	72.3	68.5	72.9
Material Balance	100.1	100.2	95.8	97.2	96.5

1 - Syncrude 2nd stage Raffinate
2 - Syncrude 3rd stage Raffinate
3 - Syncrude 4th stage Raffinate

* Results are suspect.

TABLE 21

2ND STAGE DMSO + p-TOLUENE SULPHONIC ACID EXTRACTIONS

Gas Oil Fraction	Total Nitrogen (ppm w/w as N)	Recovery (wt%)
CANMET Gas Oil Feed	4438	-
CANMET 1st Stage Raffinate	2464	86.8
CANMET 2nd Stage Raffinate (No Acid)	1903	83.0
CANMET 2nd Stage Raffinate (0.2N Acid)	1580	83.5
CANMET 2nd Stage Raffinate (2.0N Acid)	933	85.7
Suncor Gas Oil Feed	1336	-
Suncor 1st Stage Raffinate	650	97.2
Suncor 2nd Stage Raffinate (0.2N Acid)	396	96.1
Suncor 2nd Stage Raffinate (2.0N Acid)	287	94.6
Syncrude Gas Oil Feed	1473	-
Syncrude 1st Stage Raffinate	854	97.0
Syncrude 2nd Stage Raffinate (No Acid)	565	94.1
Syncrude 2nd Stage Raffinate (0.2N Acid)	489	93.5
Syncrude 2nd Stage Raffinate (2.0N Acid)	409	94.1

NOTE: All extractions were carried out at 20°C using a 1 to 1 by volume solvent to gas oil extraction ratio. The 1st stage extraction was performed using DMSO/H_2O (94:6). An aqueous 0.2N or 2.0N p-toluene sulphonic acid solution was substituted for the 6% water content in the DMSO for the 2nd stage acid extraction.

TABLE 22

MICROACTIVITY TEST RESULTS
DMSO + p-TOLUENE SULPHONIC ACID EXTRACTIONS
wt %

Run No.	Can-F	1	2	Sun-F	3	4	Syn-F	5	6
Reactor Temp. °C	530	529	529	489	489	489	490	489	489
WHSV	15.8	13.8	15.6	11.0	10.7	11.2	10.7	10.6	10.7
C/O	5.7	6.5	5.8	3.6	3.7	3.6	3.8	3.8	3.7
Reactor Effluent Gas									
Hydrogen	0.18	0.11	0.13	0.07	0.03	0.04	0.07	0.05	0.06
Methane	1.12	1.26	1.78	0.60	0.38	0.50	0.53	0.65	0.50
C_2 Hydrocarbons	1.72	2.10	1.82	1.14	0.90	1.13	0.98	1.27	1.06
C_3 Hydrocarbons	5.28	8.18	7.63	5.52	4.90	5.59	4.84	5.57	4.98
C_4 Hydrocarbons	6.46	11.28	10.6	8.75	8.36	9.12	7.38	9.10	8.44
C_5 Hydrocarbons	4.86	8.03	7.87	5.64	5.43	5.42	4.98	5.58	5.23
C_6 Hydrocarbons	1.34	1.97	2.12	1.26	1.17	1.04	0.98	1.02	1.20
C_6^+ Hydrocarbons	2.11	2.37	3.28	1.93	1.72	1.56	1.76	1.73	1.96
Product Cuts									
C_2 & lighter	3.22	4.16	4.06	1.86	1.44	1.75	1.62	2.02	1.66
C_3 & C_4	11.8	19.5	18.3	14.2	13.3	14.7	12.8	14.7	13.4
C_5^+ Gasoline (220 EP)	32.6	40.4	43.4	45.1	48.8	50.5	47.4	48.1	51.2
C_6^+ Gasoline (220 EP)	27.8	32.4	35.5	39.5	43.3	45.1	42.4	42.5	46.0
LCO (220-344°C)	22.4	18.2	17.7	21.1	21.3	19.7	21.2	20.3	19.6
Decant (344°C+)	22.8	10.6	10.2	14.1	11.1	10.2	12.3	10.7	10.1
Coke Make	7.2	7.1	6.4	3.5	4.1	3.2	4.0	4.3	4.1
Coke on Catalyst	1.2	1.1	1.1	1.0	1.1	0.9	1.1	1.1	1.1
Conversion	54.8	71.2	72.1	64.8	67.6	70.2	66.0	69.0	70.4
Material Balance	98.6	94.3	95.9	97.8	100.6	97.8	100.1	98.3	100.5

1 - CANMET Raffinate + 0.2N Acid

2 - CANMET Raffinate + 2.0N Acid

3 - Suncor Raffinate + 0.2N Acid

4 - Suncor Raffinate + 2.0N Acid

5 - Syncrude Raffinate + 0.2N Acid

6 - Syncrude Raffinate + 2.0N Acid

TABLE 23

DMSO/H_2O (94:6) TEMPERATURE EXTRACTION DATA
(1 to 1 by volume extraction ratio)

Extraction Temperature (°C)	Total Nitrogen ppm w/w as N	Recovery (wt%)
CANMET GAS OIL		
Feed	4438	-
20	2468	86.8
40	2468	82.5
60	2353	82.5
80	2406	83.8
100	2165	84.1
SYNCRUDE GAS OIL		
Feed	1473	-
20	854	97.0
40	691	94.2
60	756	98.9
80	775	94.2
100	698	97.5

TABLE 24

MICROACTIVITY TEST RESULTS
COMPARISON OF CANMET AND SYNCRUDE DMSO/H_2O (94:6) RAFFINATES
WITH VARIATION IN EXTRACTION TEMPERATURE

Run No.	Can-F	Can-R	1	2	Syn-F	Syn-R	3	4
				wt%				
Reactor Temp. °C	530	530	529	530	490	489	489	489
WHSV	15.8	14.8	14.8	14.8	10.7	11.2	11.7	10.9
Catalyst/Oil Ratio	5.7	6.1	6.1	6.1	3.8	3.6	3.4	3.6
Reactor Effluent Gas								
Hydrogen	0.18	0.16	0.13	0.15	0.07	0.06	0.06	0.05
Methane	1.12	1.34	1.01	1.16	0.53	0.66	0.42	0.57
C_2 Hydrocarbons	1.72	2.08	1.79	1.85	0.98	1.22	0.92	1.23
C_3 Hydrocarbons	5.28	6.84	5.74	6.45	4.84	5.34	4.56	5.12
C_4 Hydrocarbons	6.46	9.00	7.59	8.91	7.38	8.66	7.64	8.50
C_5 Hydrocarbons	4.86	6.52	5.21	6.82	4.98	5.36	5.14	4.35
C_6 Hydrocarbons	1.34	1.59	1.16	1.91	0.98	1.02	1.12	0.99
C_6+ Hydrocarbons	2.11	1.97	1.85	3.04	1.76	1.44	1.83	2.99
Product Cuts								
C_2 & lighter	3.22	4.04	3.21	3.45	1.62	1.96	1.46	1.83
C_3 & C_4	11.8	15.8	13.3	15.4	12.8	14.0	12.2	13.6
C_5+ Gasoline (220 EP)	32.6	35.3	38.0	43.8	47.4	49.0	49.3	53.1
C_6+ Gasoline (220 EP)	27.8	28.8	32.8	36.9	42.4	44.0	44.2	48.6
LCO (220-344°C)	22.4	20.2	21.2	17.6	21.2	20.2	21.4	19.4
Decant (344°C+)	22.8	17.2	17.7	12.9	12.3	10.6	12.3	8.2
Coke Make	7.2	7.4	6.6	7.0	4.0	4.3	3.3	3.9
Coke on Catalyst	1.2	1.2	1.1	1.1	1.1	1.2	1.0	1.1
Conversion	54.8	62.6	61.2	69.5	66.0	69.2	66.3	72.5
Material Balance	98.6	96.7	98.8	98.9	100.1	100.2	100.3	96.5

1 - CANMET DMSO/H_2O (94:6) Raffinate @ 80°C
2 - CANMET DMSO/H_2O (94:6) Raffinate @ 100°C
3 - Syncrude DMSO/H_2O (94:6) Raffinate @ 80°C
4 - Syncrude DMSO/H_2O (94:6) Raffinate @ 100°C

TABLE 25

HPLC APROTIC SOLVENT ANALYSES OF GAS OIL RAFFINATES

Raffinate	Residual DMF (ppm v/v as DMF)*	Residual NMP (ppm v/v as NMP)**	Residual DMSO (ppm v/v as DMSO)***
CANMET DMF/H_2O (90:10)	1	-	-
Suncor DMF/H_2O (90:10)	1	-	-
Syncrude DMF/H_2O (90:10)	19	-	-
CANMET NMP/H_2O (90:10)	-	1	-
Suncor NMP/H_2O (90:10)	-	131	-
Syncrude NMP/H_2O (90:10)	-	295	-
CANMET DMSO/H_2O (94:6) (1/1 Solvent to Oil)	-	-	4
Suncor DMSO/H_2O (94:6) (1/1 Solvent to Oil)	-	-	11
Syncrude DMSO/H_2O (94:6) (1/1 Solvent to Oil)	-	-	4
CANMET DMSO/H_2O (94:6) (2/1 Solvent to Oil)	-	-	7
Suncor DMSO/H_2O (94:6) (2/1 Solvent to Oil)	-	-	13
Syncrude DMSO/H_2O (94:6) (2/1 Solvent to Oil)	-	-	50
CANMET 2nd Stage DMSO + 2N Acid (94:6)	-	-	3
Suncor 2nd Stage DMSO + 2N Acid (94:6)	-	-	8
Syncrude 2nd Stage DMSO + 2N Acid (94:6)	-	-	61
CANMET 3rd Stage DMSO/H_2O (94:6)	-	-	2
CANMET 4th Stage DMSO/H_2O (94:6)	-	-	3

* 1 ppm v/v DMF - equals approx. 0.2 ppm w/w as N

** 1 ppm v/v NMP - equals approx. 0.16 ppm w/w as N

*** 1 ppm v/v DMSO - equals 0.0 ppm w/w as N

TABLE 26

MICROACTIVITY TEST RESULTS
DMSO CONTAMINATED RAFFINATES (1% DMSO)
wt%

Run No.	Can-R	1	Sun-R	2	Syn-R	3
Reactor Temp. °C	530	530	489	489	489	489
WHSV	14.8	15.6	10.2	10.8	11.2	10.2
C/O	6.1	5.8	3.9	3.7	3.6	3.9
Reactor Effluent Gas						
Hydrogen	0.16	0.13	0.06	0.04	0.06	0.05
Methane	1.34	1.62	0.66	0.72	0.66	0.82
C_2 Hydrocarbons	2.08	2.12	1.30	1.14	1.22	1.27
C_3 Hydrocarbons	6.84	7.27	6.02	5.21	5.34	5.41
C_4 Hydrocarbons	9.00	9.40	9.69	8.45	8.66	8.83
C_5 Hydrocarbons	6.52	6.80	5.95	5.01	5.36	5.90
C_6 Hydrocarbons	1.59	1.81	1.14	0.80	1.02	1.12
C_6^+ Hydrocarbons	1.97	2.69	1.70	1.08	1.44	1.44
Product Cuts						
C_2 & lighter	4.04	4.87	2.13	2.27	1.96	2.38
C_3 & C_4	15.8	16.7	15.7	13.7	14.0	14.2
C_5^+ Gasoline (220 EP)	35.3	36.9	49.1	50.2	49.0	47.4
C_6^+ Gasoline (220 EP)	28.8	30.1	43.1	45.2	44.0	41.5
LCO (220-344°C)	20.2	19.6	19.3	19.7	20.2	20.6
Decant (344°C+)	17.2	14.6	9.8	10.6	10.6	11.3
Coke Make	7.4	7.5	3.9	3.6	4.3	4.2
Coke on Catalyst	1.2	1.3	1.0	1.2	1.2	1.3
Conversion	62.6	65.9	70.8	69.7	69.2	68.2
Material Balance	96.7	96.5	96.5	97.7	100.2	97.4

1 - CNAMET DMSO Raffinate + 1% DMSO

2 - Suncor DMSO RAffinate + 1% DMSO

3 - Syncrude DMSO Raffinate + 1% DMSO

TABLE 27

COMPOSITIONAL CHARACTERIZATION OF EXTRACTS
% by wt

	1st Stage DMSO CANMET Extract	1st Stage DMSO Suncor Extract	1st Stage DMSO Syncrude Extract	2nd Stage DMSO Syncrude Extract
L.C. Separation				
Saturates & Aromatics	99.0	99.0	98.8	99.0
Polars	1.0	1.0	1.2	1.0
Recovery	98.5	100.2	103.1	104.5
Elemental Analyses				
Carbon	85.18	87.99	87.72	87.99
Hydrogen	7.32	8.21	8.56	8.21
Nitrogen	2.92	2.27	2.25	2.27
Sulfur	4.04	1.45	1.16	1.13
Oxygen (By difference)	0.54	0.08	0.31	0.40
Other Analyses				
Basic Nitrogen as N	0.59	0.07	0.06	0.06
Water as H_2O	0.17	0.05	0.08	0.13
Mass Spec Analyses				
Nitrogen Compounds	(43.5)	(31.2)	(37.9)	(27.9)
Pyridines	-	-	-	0.5
Indoles	0.9	0.2	-	-
Quinolines	5.2	1.3	0.8	1.4
Carbazoles	15.6	15.2	14.2	14.1
Acridines	12.7	11.1	17.4	8.4
Benzcarbazoles	5.2	2.7	4.8	3.3
Benzacridines	3.9	0.7	0.7	0.2
Hydrocarbons	(45.9)	(63.6)	(50.7)	(67.9)
Paraffins	-	0.4	-	-
Cycloparaffins	-	5.0	0.6	-
Monoaromatacs	3.9	5.7	8.7	8.0
Diaromatics	6.4	2.5	5.4	4.8
Triaromatics	12.9	24.6	14.3	32.0
Tetraaromatics	8.5	16.8	15.2	16.0
Pentaaromatacs	14.0	8.4	6.5	7.0
Polyaromatics	0.2	0.2	-	0.1
Other, i.e. Thiophenes, Phenols, H_2O, etc.	10.6	5.2	11.4	4.2
Infra-Red Analyses				
NH Content	12.57	10.73	14.62	8.66
OH Content	1.30	0.25	0.20	0.30
-C=O Content (acidic)	0.41	0.91	0.81	0.97

FIGURE 1

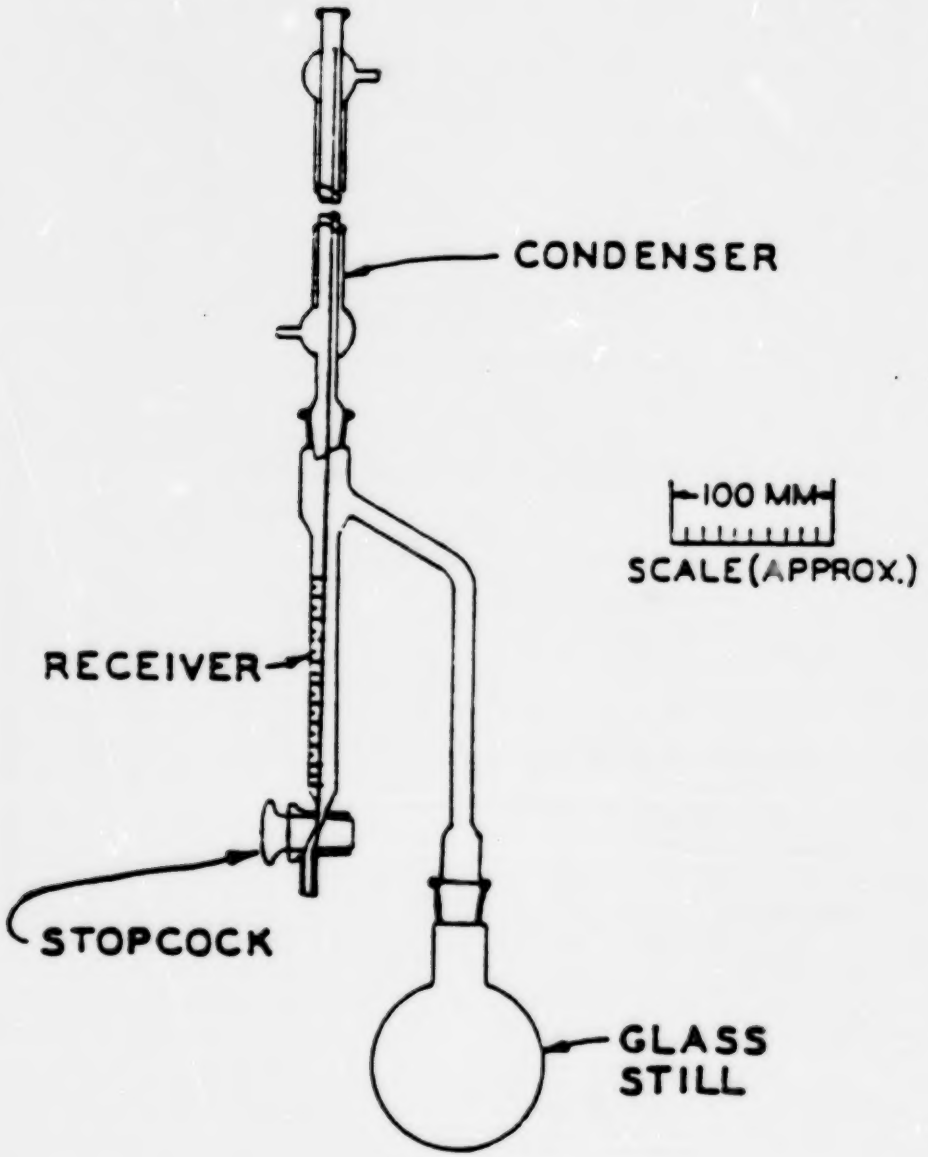


FIG. 1 Typical Assembly with Glass Still (Dean and Stark).

FIGURE 2

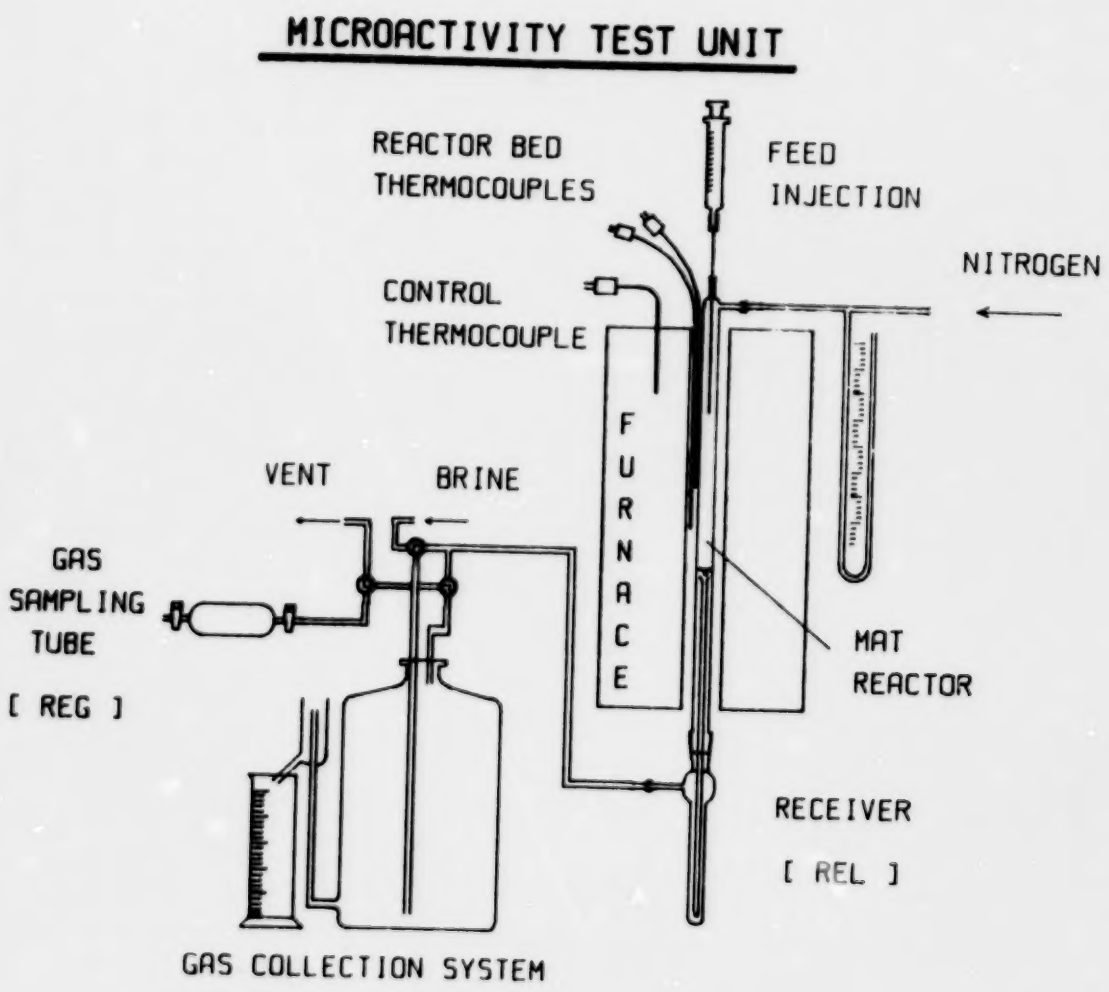

MICROACTIVITY TEST UNIT
REACTOR BED THERMOCOUPLES
FEED INJECTION
NITROGEN
CONTROL THERMOCOUPLE
FURNACE
VENT
BRINE
GAS SAMPLING TUBE
[REG]
MAT REACTOR
RECEIVER
[REL]
GAS COLLECTION SYSTEM

FIGURE 3

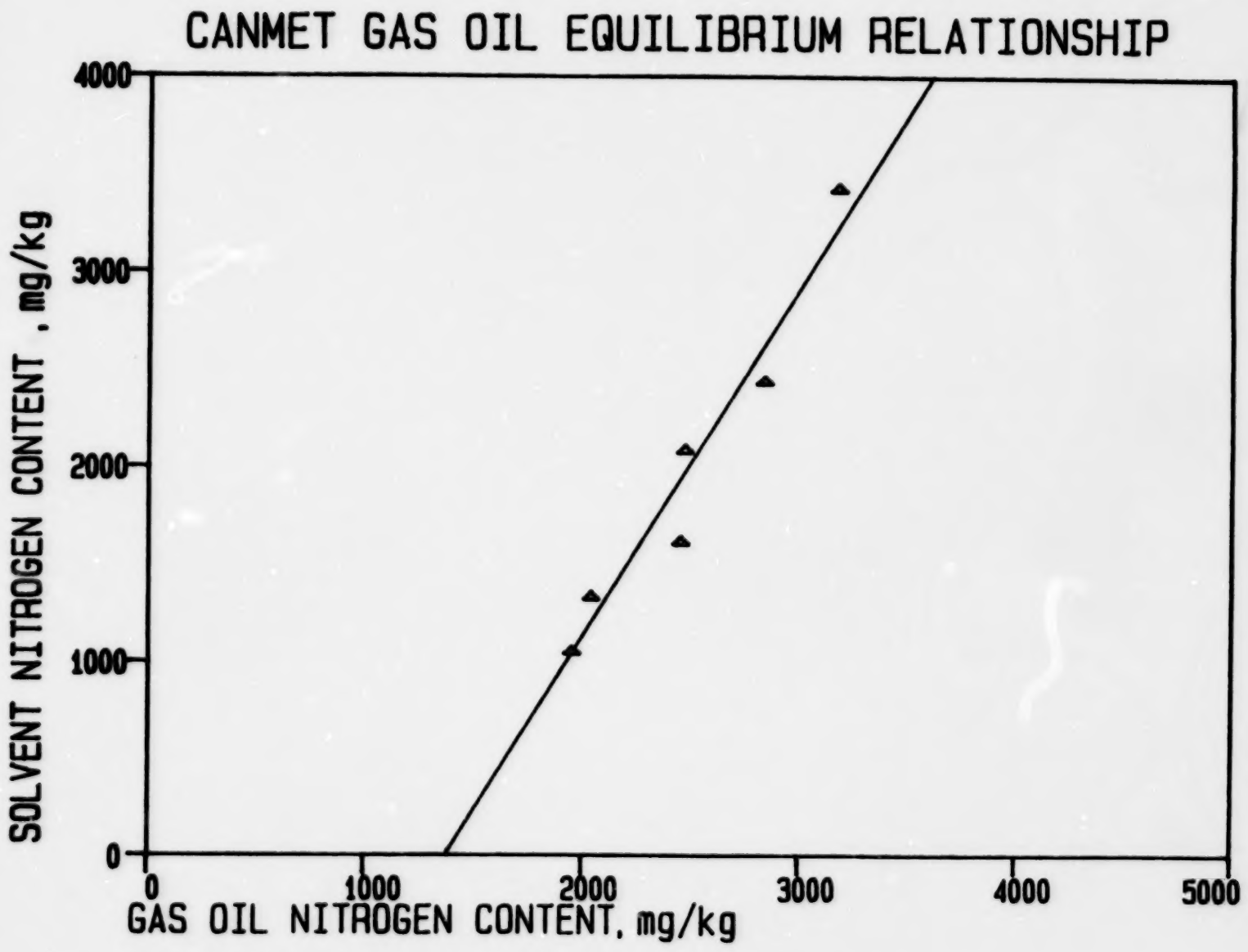

CANMET GAS OIL EQUILIBRIUM RELATIONSHIP
SOLVENT NITROGEN CONTENT, mg/kg
4000
3000
2000
1000
0
0
1000
2000
3000
4000
5000
GAS OIL NITROGEN CONTENT, mg/kg

FIGURE 4

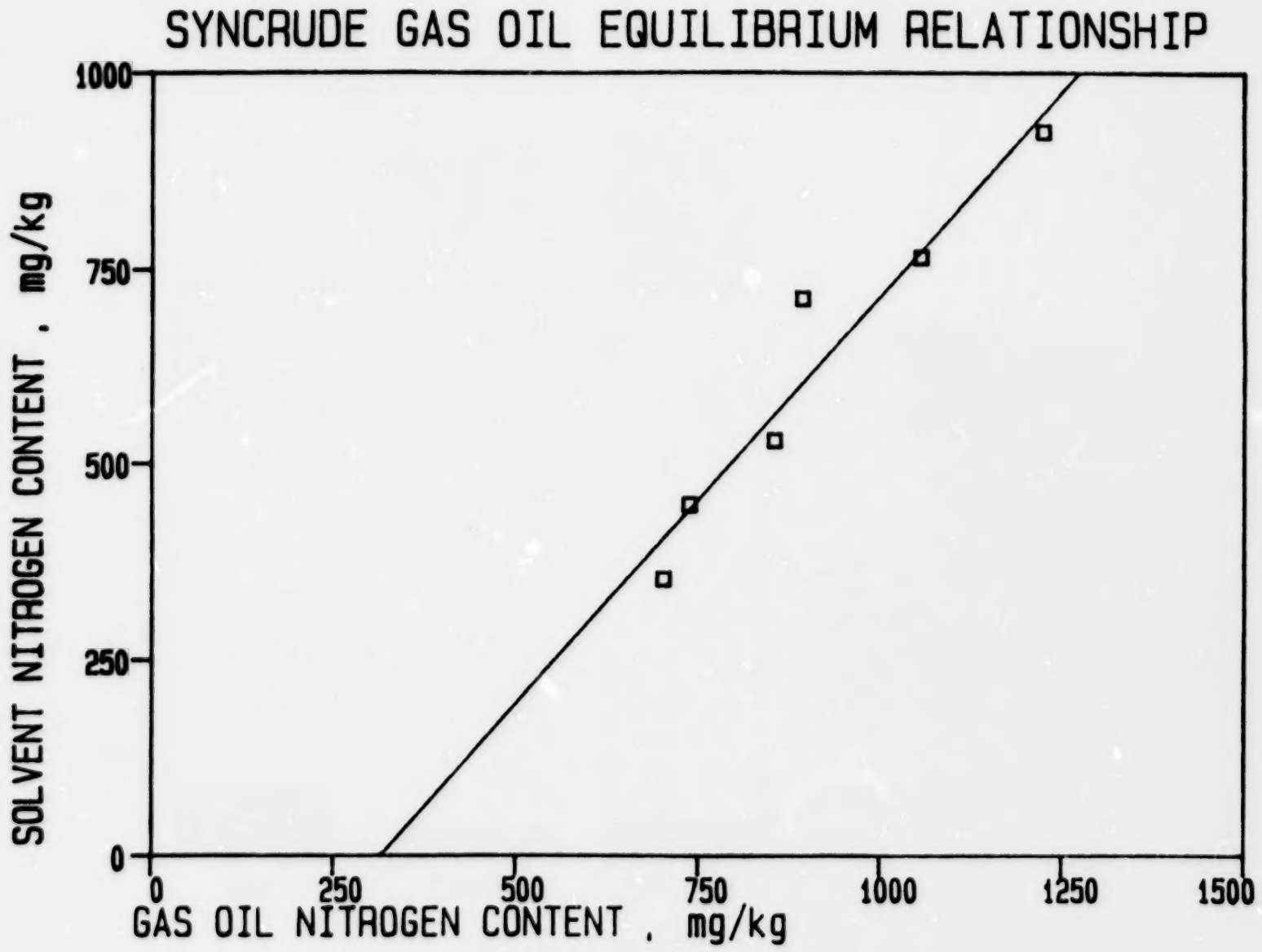

SYNCRUDE GAS OIL EQUILIBRIUM RELATIONSHIP
SOLVENT NITROGEN CONTENT , mg/kg
1000
750
500
250
0
0
250
500
750
1000
1250
1500
GAS OIL NITROGEN CONTENT , mg/kg

FIGURE 5

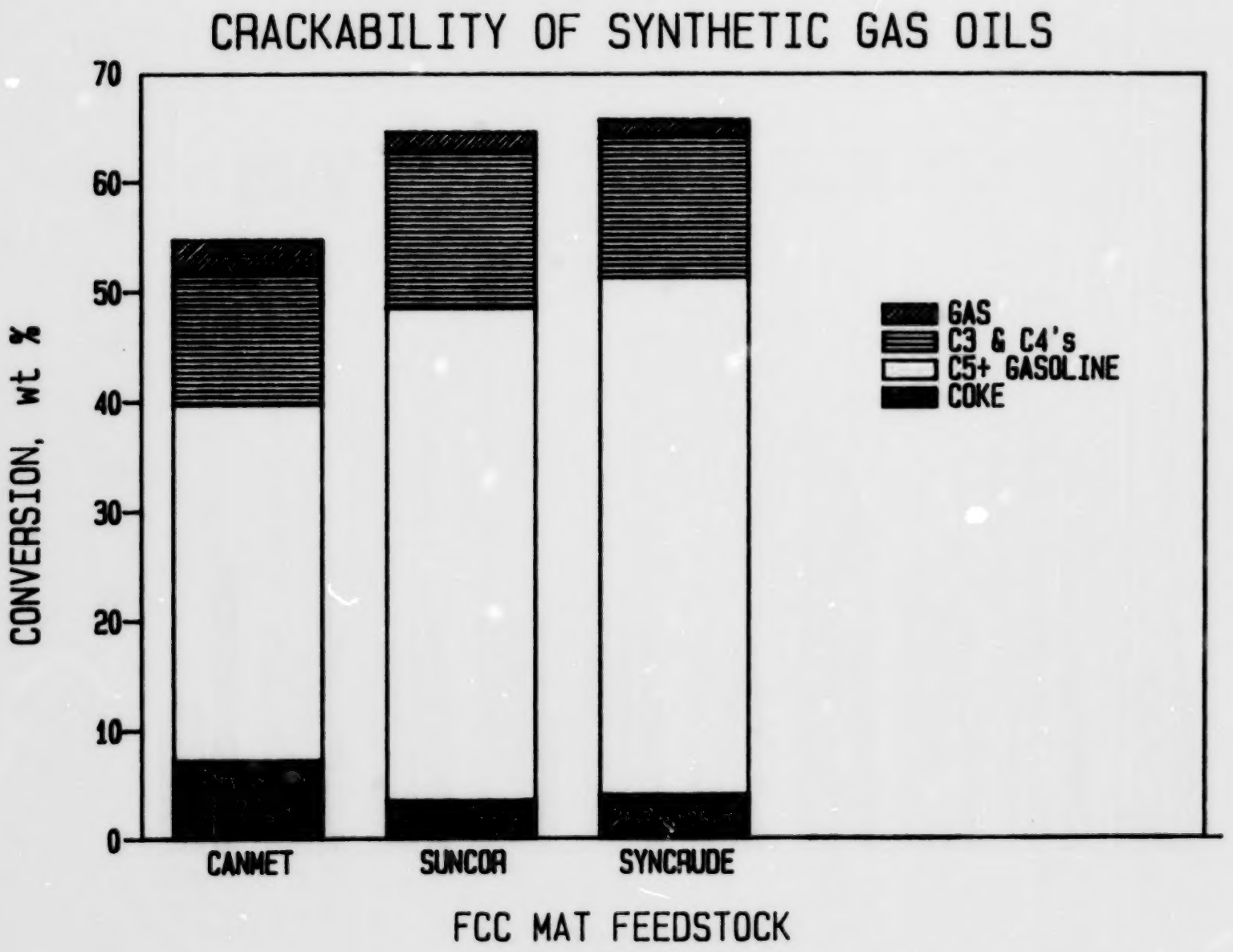

CRACKABILITY OF SYNTHETIC GAS OILS
CONVERSION, wt %
70
60
50
40
30
20
10
0
GAS
C3 & C4's
C5+ GASOLINE
COKE
CANMET
SUNCOR
SYNCRUDE
FCC MAT FEEDSTOCK

FIGURE 6

(see Table 28 for bar graph glossary)

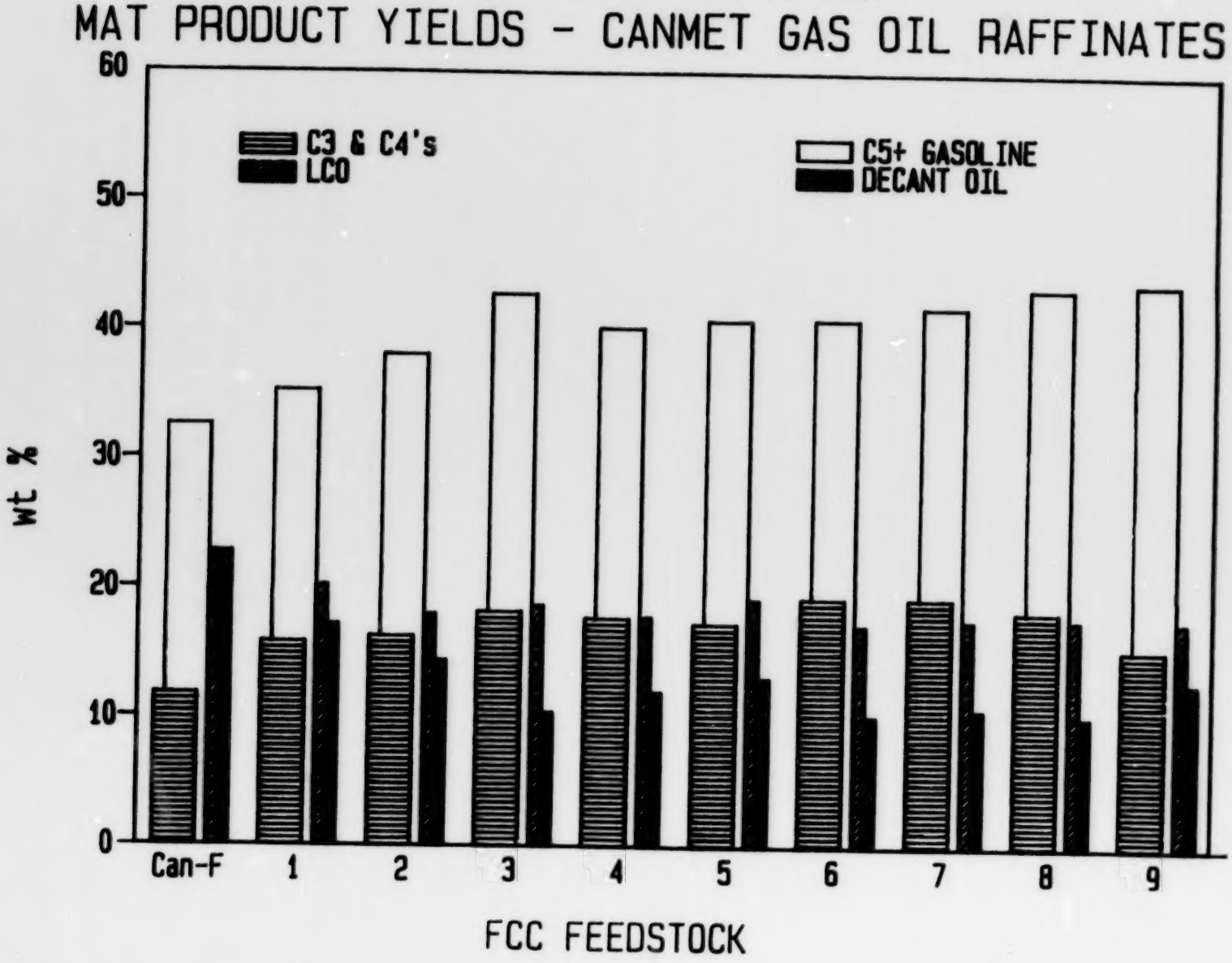


TABLE 28

GLOSSARY FOR FIGURES 6, 7 AND 8

FIGURE 6 (MAT Product Yields - CANMET Gas Oil Raffinates)

1. DMSO/H_2O (94:6) (Averaged) @ 20°C Extraction Temperature and Solvent to Gas Oil Extraction Ratio 1/1
2. DMF/H_2O (90:10) @ 20°C and 1/1
3. NMP/H_2O (90:10) @ 20°C and 1/1
4. DMSO/H_2O @ Solvent to Oil Extraction Ratio 2/1 and 20°C
5. 2nd Stage DMSO raffinate @ 20°C and 1/1
6. 3rd Stage DMSO raffinate @ 20°C and 1/1
7. 4th Stage DMSO raffiante @ 20°C and 1/1
8. Can-R extracted with DMSO/2N aqueous p-toluene sulphonic acid (94:6) @ 20°C and 1/1
9. DMSO/H_2O (94:6) @ 100°C and 1/1

FIGURE 7 (MAT Product Yields - Suncor Gas Oil Raffinates)

1. DMSO/H_2O (94:6) (Averaged) @ 20°C Extraction Temperature and Solvent to Gas Oil extraction Ratio 1/1
2. DMF/H_2O (90:10) @ 20°C and 1/1
3. NMP/H_2O (90:10) @ 20°C and 1/1
4. Sun-R extracted with DMSO/2N aqueous p-toluene sulphonic acid (94:6) @ 20°C and 1/1
5. DMSO/H_2O (94:6) Solvent to Gas Oil Extraction Ratio 2/1 @ 20°C

FIGURE 8 MAT Product Yields - Syncrude Gas Oil Raffinates)

1. DMSO/H_2O (94:6) (Averaged) @ 20°C Extraction Temperature and Solvent to Gas Oil Extraction Ratio 1/1
2. DMF/H_2O (90:10) @ 20°C and 1/1
3. NMP/H_2O (90:10) @ 20°C and 1/1
4. DMSO/H_2O (94:6) Solvent to Gas Oil Extraction Ratio 2/1 @ 20°C
5. 2nd Stage DMSO raffinate @ 40°C and 1/1
6. 3rd Stage DMSO raffinate @ 40°C and 1/1
7. 4th Stage DMSO raffinate @ 40°C and 1/1
8. Syn-R extracted with DMSO/2N aqueous p-toluene sulphnoic acid (94:6) @ 20°C and 1/1
9. DMSO/H_2O (94:6) @ 100°C and 1/1

FIGURE 7

(see Table 28 for bar graph glossary)

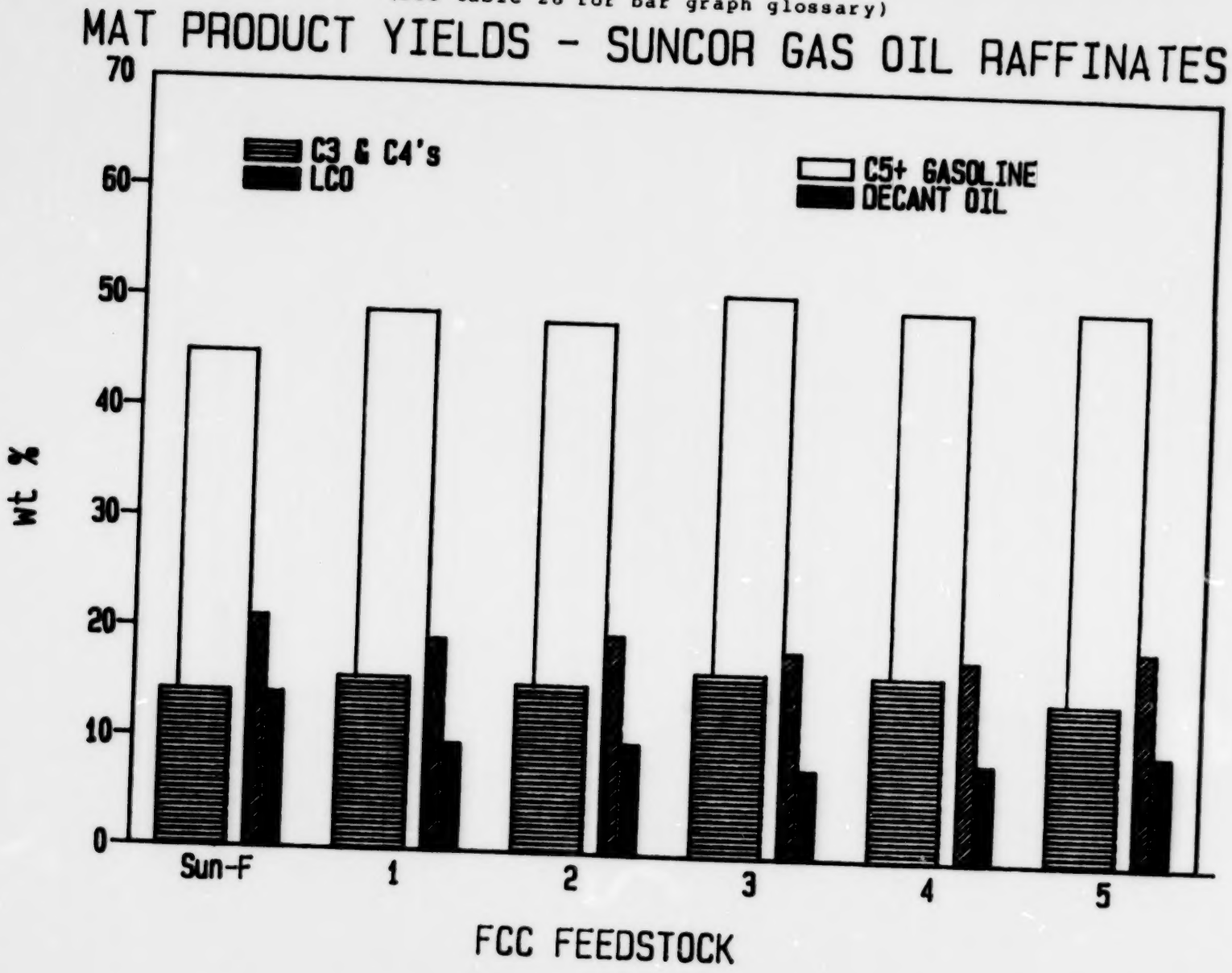
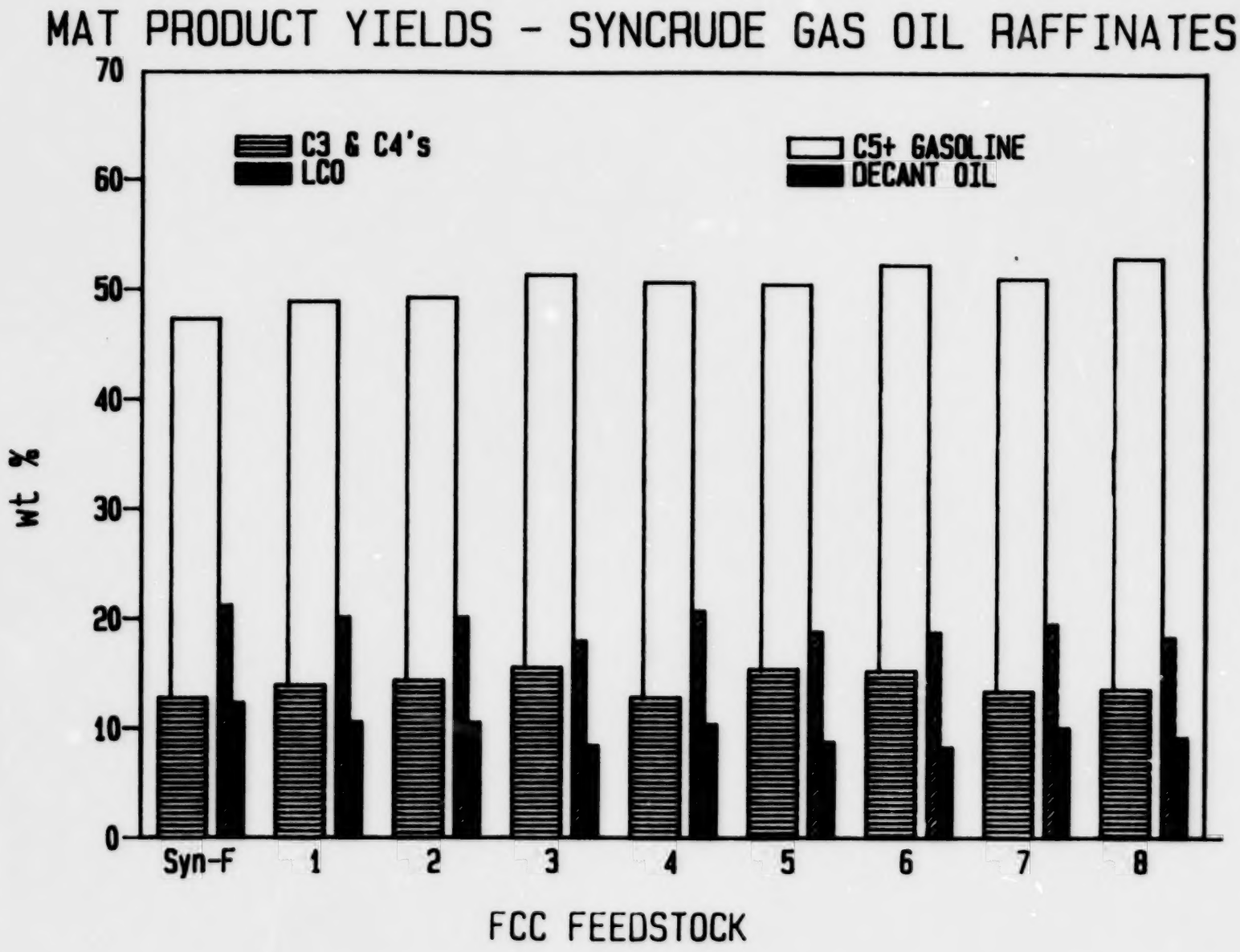


FIGURE 8

(see Table 28 for bar graph glossary)



APPENDUM

CONTENTS OF APPENDUM

APPENDUM A

Determination of DMF, DMSO and NMP in Gas Oil by HPLC

List of Figures

FIGURE A-1	DIAGRAM OF HPLC ANALYZER
FIGURE A-2	DIAGRAM OF COLUMN SWITCHING CONFIGURATION
FIGURE A-3	CALIBRATION CURVES FOR DMF, NMP AND DMSO
FIGURE A-4a	HPLC CHROMATOGRAMS OF DMF CALIBRATION STANDARDS
FIGURE A-4b	HPLC CHROMATOGRAMS OF GAS OILS CONTAINING DMF
FIGURE A-4c	HPLC COMARISON OF CANMET GAS OIL AND CANMET RAFFINATE
FIGURE A-4d	HPLC CHROMATOGRAMS OF GAS OILS AND POLAR SOLVENTS
FIGURE A-4e	HPLC CHROMATOGRAMS OF GAS OIL RAFFINATES FROM 2N ACID + DMSO EXTRACTION
FIGURE A-4f	HPLC CHROMATOGRAMS OF RAFFINATES FROM 2:1 DMSO/H_2O (94:6) EXTRACTION
FIGURE A-4g	HPLC CHROMATOGRAMS OF GAS OIL RAFFINATES FROM NMP EXTRACTION
FIGURE A-4h	HPLC CHROMATOGRAMS OF GAS OIL RAFFINATES FROM DMSO/H_2O EXTRACTION

APPENDUM B

SELECTIVE EXTRACTION OF NITROGEN CONSTITUENTS FROM FLUID CATALYTIC CRACKING FEEDSTOCKS by L. E. Curts

List of Tables

TABLE B-1	PDA AND SYNCRUDE FEEDSTOCKS CHARACTERIZATION
TABLE B-2	HIGH RESOLUTION MASS SPECTROMETER ANALYSES OF PDA AND SYNCRUDE FEEDSTOCKS
TABLE B-3	TOTAL NITROGEN RESULTS OF PDA AND SYNCRUDE FEEDSTOCKS

APPENDIX A

DETERMINATION OF DMF, DMSO AND NMP IN GAS OIL BY HPLC

1. OUTLINE OF METHOD

The sample is diluted with cyclohexane (1:1 v/v) and analysed on a high performance liquid chromatograph (HPLC) using a C-18 reverse phase column. The method utilizes isocratic elution with 20% acetonitrile and 80% water to separate the polar solvent from the gas oil. Once the solvent of interest is eluted, the column is back-flushed with 100% THF and then with 100% acetonitrile to elute the retained gas oil from the column. The components of interest are quantitatively determined using external calibration and monitored at 220 nm in the UV.

2. SCOPE

This method covers the analysis of specific residual polar solvents present in gas oils. These solvents include N,N-dimethylformamide (DMF), dimethylsulfoxide (DMSO) and N-methyl-2-pyrolidone (NMP). The method is applicable for the determination of a single solvent in gas oils. Because the samples are viscous, they must be diluted with cyclohexane at 1:1 v/v prior to injection. This dilution must be accounted for in the final calculation. The detection limit of the method is better than 25 ppm by volume.

3. APPARATUS

3.1 HPLC CHROMATOGRAPH

3.1.1 SOLVENT DELIVERY SYSTEM: Any system equipped with ternary solvent delivery capability is suitable for this application. A proportional valve solvent delivery system provides simpler operation.

3.1.2 DETECTOR: A variable wavelength UV detector or single wavelength detector capable of monitoring at 220 nm.

3.1.3 INJECTOR: A variable volume or fixed loop injector capable of injecting 50 L sample.

3.1.4 SWITCHING VALVE FOR COLUMN BACKFLUSH: Any HPLC six port valve.

3.1.5 INTEGRATOR: Any integrator capable of integrating correctly the areas of unresolved peaks.

3.1.6 COLUMNS: Reverse phase C-18 column with guard column, e.g. DuPont Zorbax ODS cartridge column (Part No. 820668.932) and guard column (Part No. 820674.902) in a cartridge column fitting kit (Part No. 820678.901).

3.1.7 SOLVENT FILTRATION SYSTEM: e.g. Millipore Catalog 04700,20,30.

3.1.8 SOLVENT FILTERS: Millipore 0.20 FGLP and 0.22 GSWP.

4. REAGENTS

4.1 HPLC GRADE SOLVENTS

4.1.1 ACETRONITRILE: Filtered through 0.20 µm Millipore FGLP membrane.

4.1.2 WATER: Filtered through 0.22 µm Millipore GSWP membrane. Do not use prolonged storage water. A freshly purified water from Milli-Q water purification system or comparable equipment is recommended.

4.1.3 TETRAHYDROFURAN (Non-preservative): Filtered through 0.22 m Millipore membrane.

4.2 POLAR SOLVENTS

4.2.1 DIMETHYLSULFOXIDE (DMSO): Reagent grade 99%+

4.2.2 N,N-DIMETHYLFORMAMIDE (DMF): Reagent grade 99%+

4.2.3 N-METHYL-2-PYROLIDONE (NM): Reagent grade 99%+

4.3 PURGE GAS: Flow regulated zero grade Helium

5. PROCEDURE

5.1 Set up instrumentation as per Figure A-1 and Figure A-2 and begin degassing eluents.

5.2 Set up HPLC solvent delivery parameters as follows:

Time min	Acetonitrile vol%	Water vol%	TFH vol%	Column Mode
0	20	80	0	Forward
7	0	0	100	Backflush
14	100	0	0	Backflush
20	20	80	0	Forward

5.3 Turn on HPLC solvent delivery system and let equilibrate. Turn on UV detector and set monitor wavelength at 220 nm and let warm up for at least 10 min.

Perform a blank run to clean any contaminants from column.

5.4 Inject 50 L of appropriate calibration standard. Immediately upon injection start sequence. Once the elution cycle is completed, let equilibrate at zero time for 5 minutes.

5.5 Repeat step 5.4 for additional calibration standards and construct a calibration curve for each polar solvent to be measured as shown in Figure A-3.

5.6 Perform sample analyses as per step 5.4 (sample should be diluted with cyclohexane at 1:1 v/v before injection).

8. CALCULATION

Polar Solvent Concentration, ppm v/v = As x Ks x 2

Ks (Calibration factor of Polar Solvent) = Ci/Ai

Ks = Calibration factor of polar solvents
Ci = Polar solvent concentration (ppm v/v) in standard
Ai = Area count of polar solvent in standard
As = Area count of polar solvent in sample

FIGURE A-1

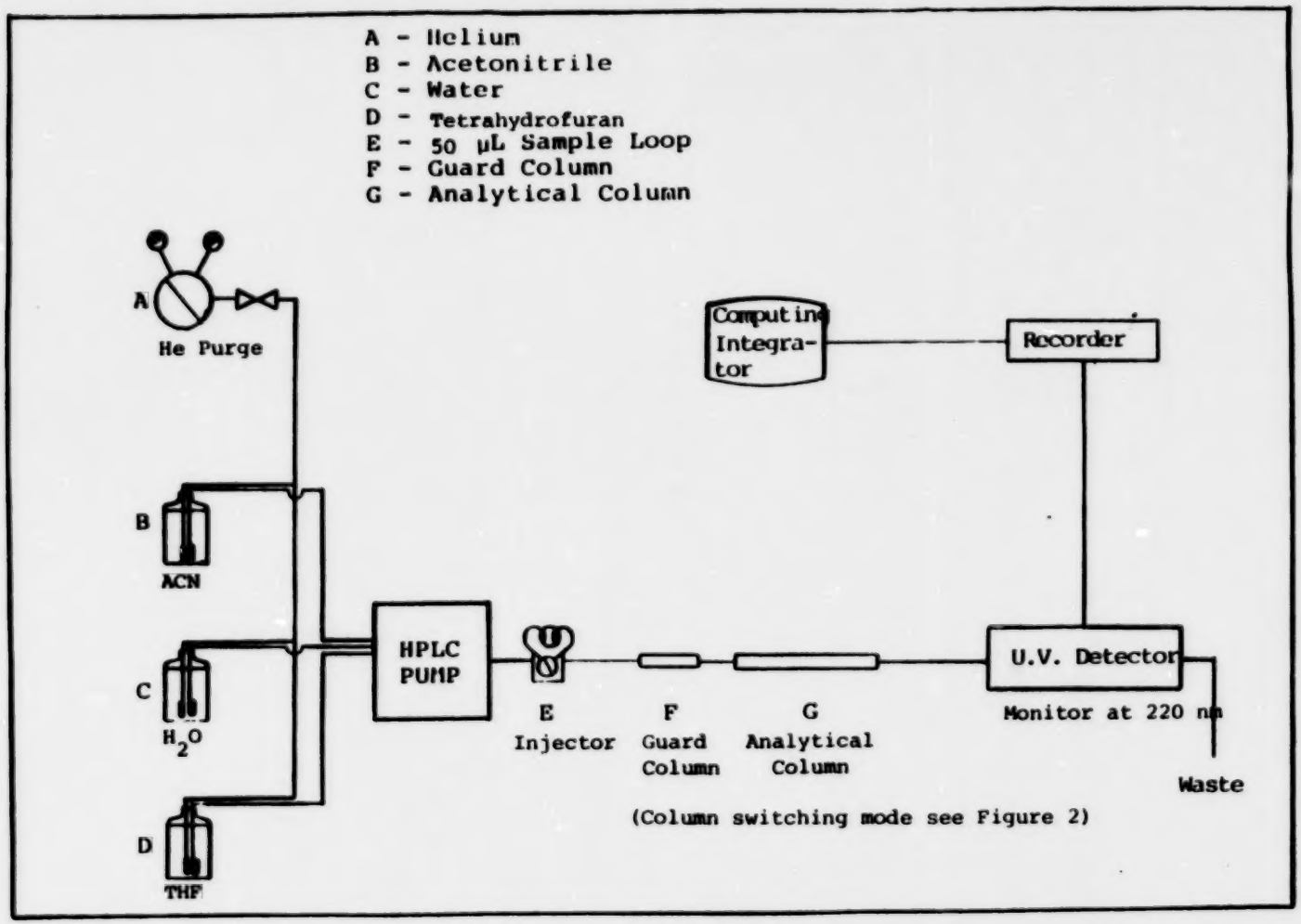
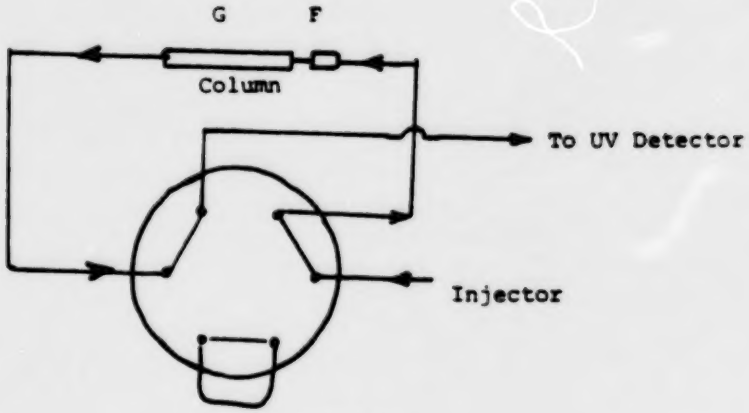
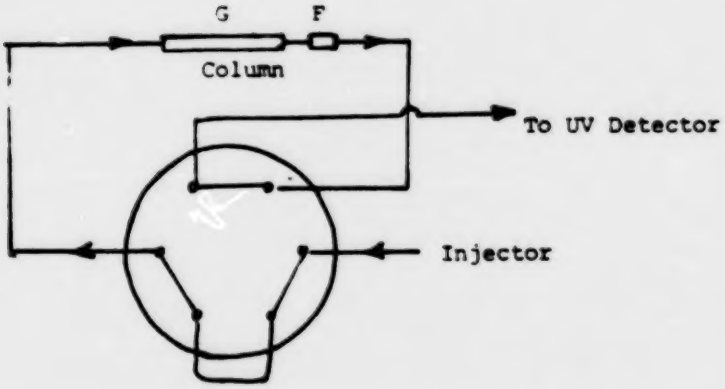

A - Helium
B - Acetonitrile
C - Water
D - Tetrahydrofuran
E - 50 µL Sample Loop
F - Guard Column
G - Analytical Column
A
He Purge
B
ACN
C
H_2O
D
THF
HPLC PUMP
Computing Integrator
Recorder
U.V. Detector
Monitor at 220 nm
E
Injector
F
Guard Column
G
Analytical Column
Waste
(Column switching mode see Figure 2)

FIGURE A-2: COLUMN SWITCHING CONFIGURATION



INJECTION MODE



BACKFLUSH MODE

FIGURE A-3

CALIBRATION CURVES OF DMF,NMP, AND DMSO
ISOCRATIC 80% WATER AND 20% ACETONITRILE
UV MONITOR AT 220 nm

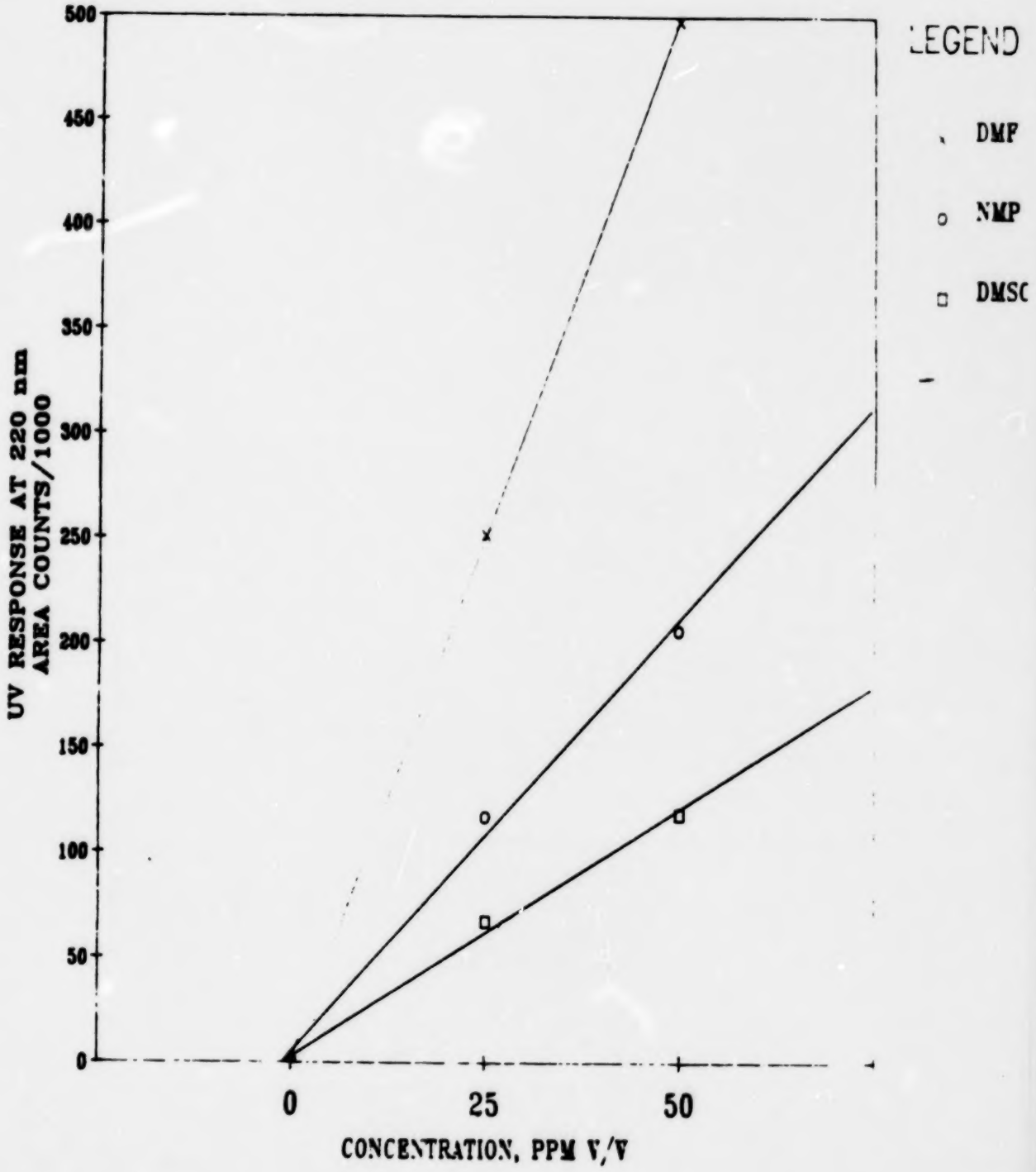


FIGURE A-4a: HPLC CHROMATOGRAMS OF DMF CALIBRATION STANDARDS

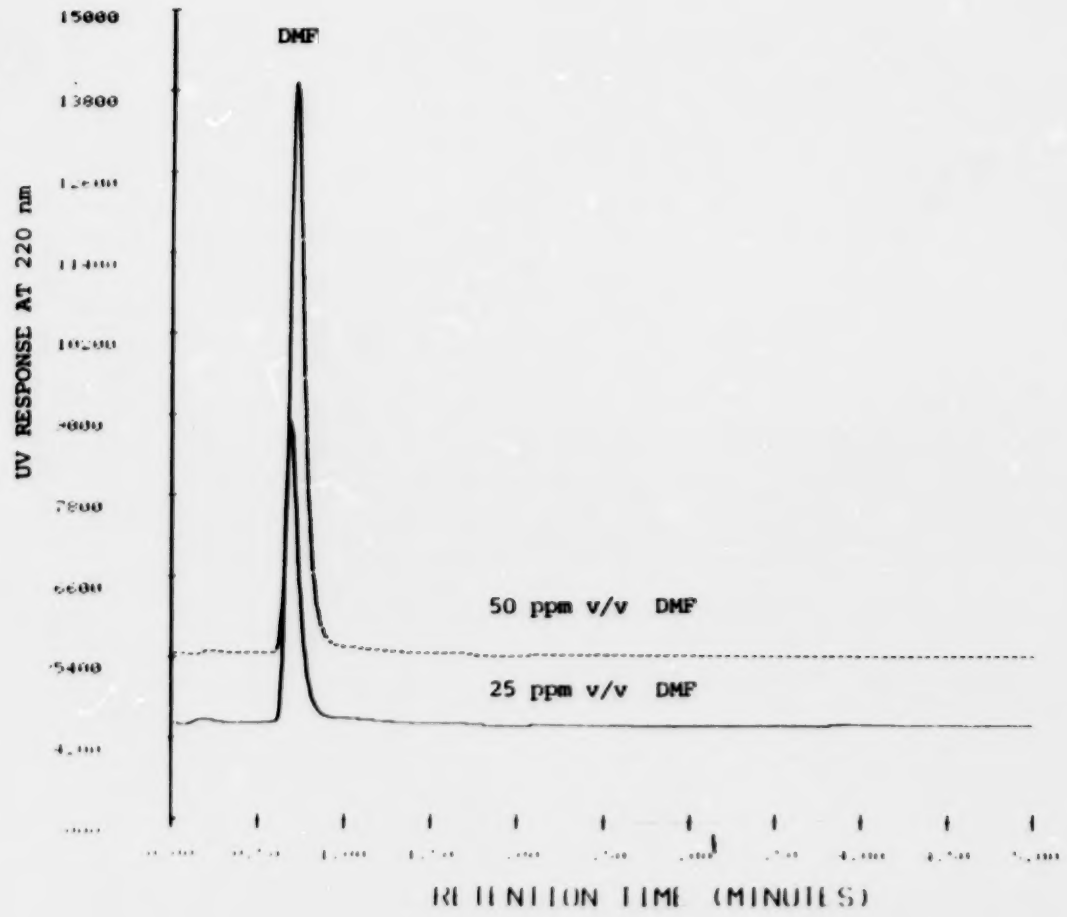


FIGURE A-4b: HPLC CHROMATOGRAMS OF GAS OIL CONTAINING DMF

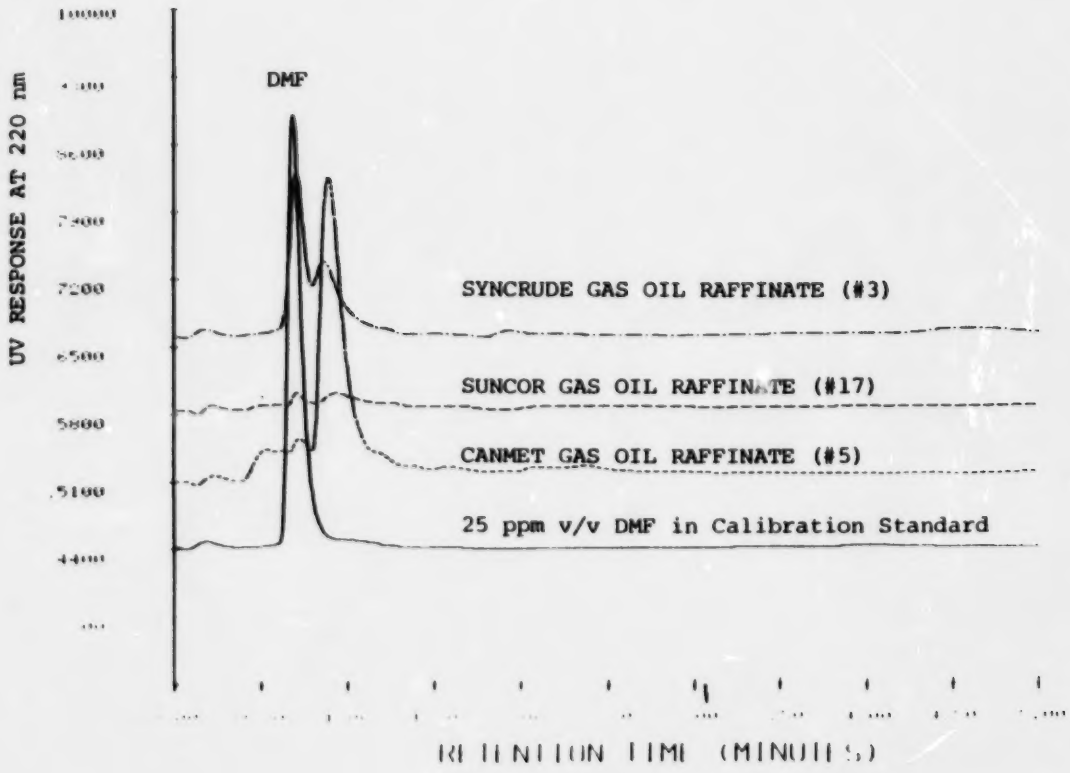

DMF
SYNCRUDE GAS OIL RAFFINATE (#3)
SUNCOR GAS OIL RAFFINATE (#17)
CANMET GAS OIL RAFFINATE (#5)
25 ppm v/v DMF in Calibration Standard
UV RESPONSE AT 220 nm
RETENTION TIME (MINUTES)

FIGURE A-4c: HPLC COMPARISON OF CANMET GAS OIL AND CANMET RAFFINATE

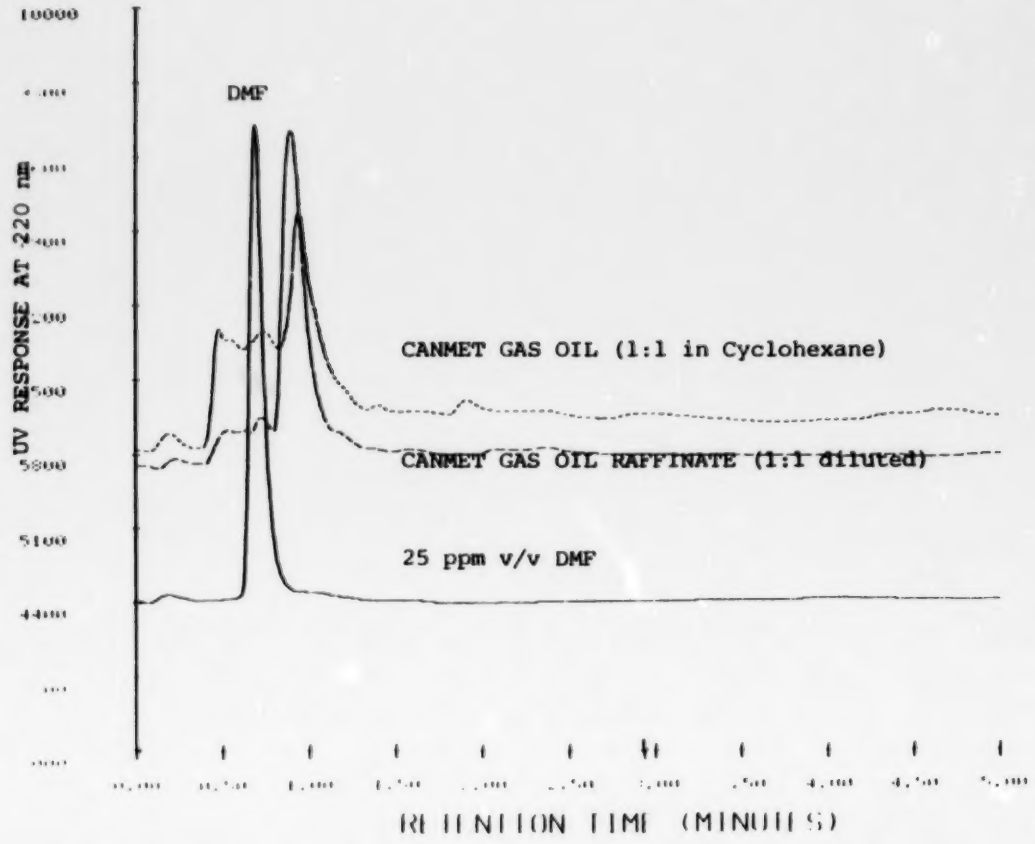

DMF
CANMET GAS OIL (1:1 in Cyclohexane)
CANMET GAS OIL RAFFINATE (1:1 diluted)
25 ppm v/v DMF
UV RESPONSE AT 220 nm
RETENTION TIME (MINUTES)

FIGURE A-4d: HPLC CHROMATOGRAMS OF GAS OILS AND POLAR SOLVENTS

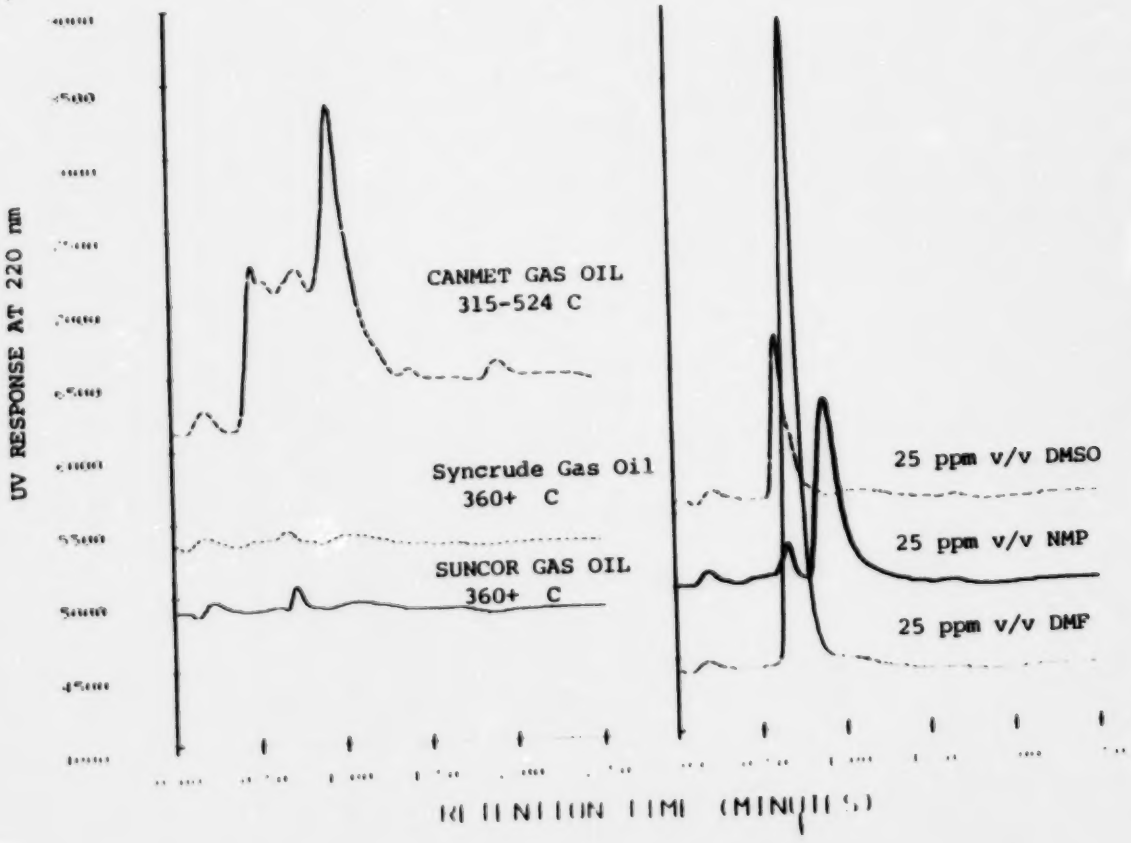


FIGURE A-4e: HPLC CHROMATOGRAMS OF GAS OIL RAFFINATES FROM 2N Acid + DMSO EXTRACTION

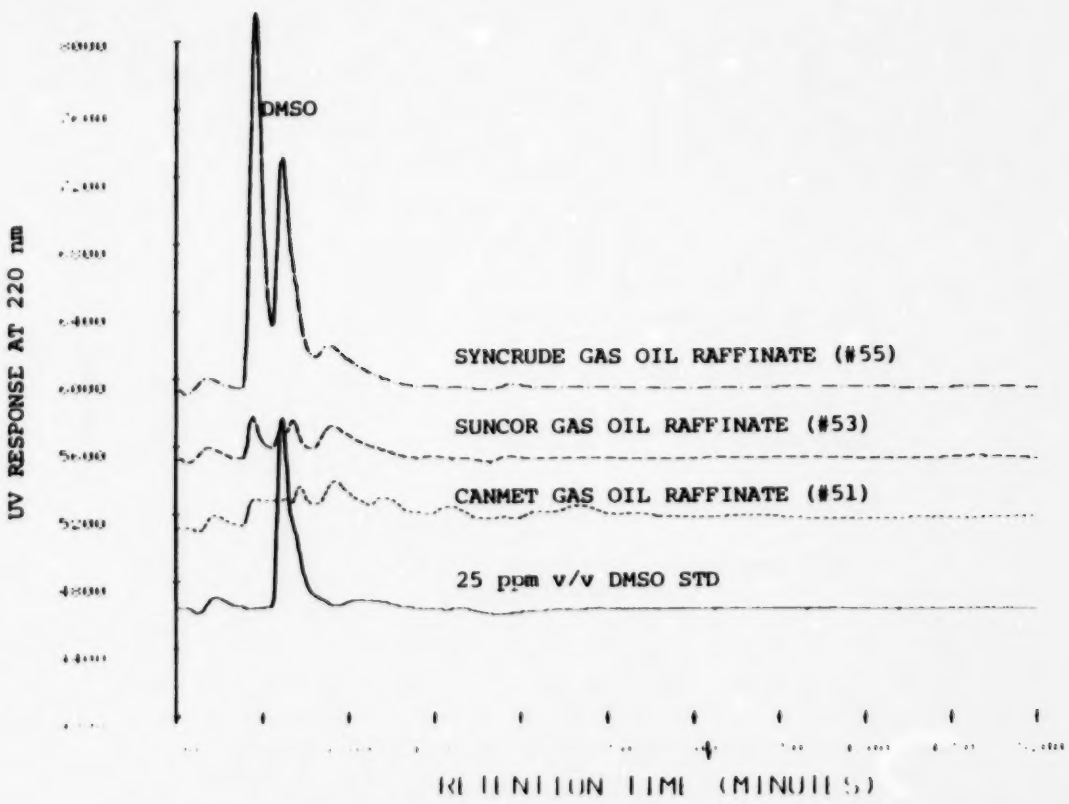

DMSO
SYNCRUDE GAS OIL RAFFINATE (#55)
SUNCOR GAS OIL RAFFINATE (#53)
CANMET GAS OIL RAFFINATE (#51)
25 ppm v/v DMSO STD
UV RESPONSE AT 220 nm
RETENTION TIME (MINUTES)

FIGURE A-4f: HPLC CHROMATOGRAMS OF RAFFINATES FROM 2:1 DMSO/H_2O (94:6) EXTRACTION

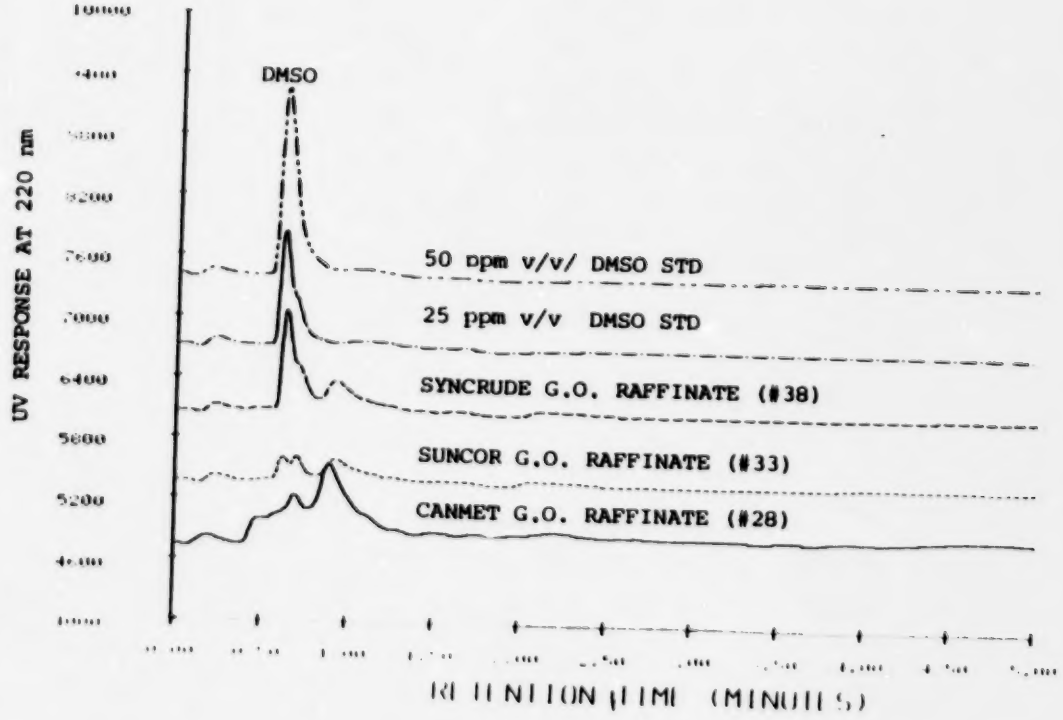


FIGURE A-4g: HPLC CHROMATOGRAMS OF GAS OIL RAFFINATES FROM NMP EXTRACTION

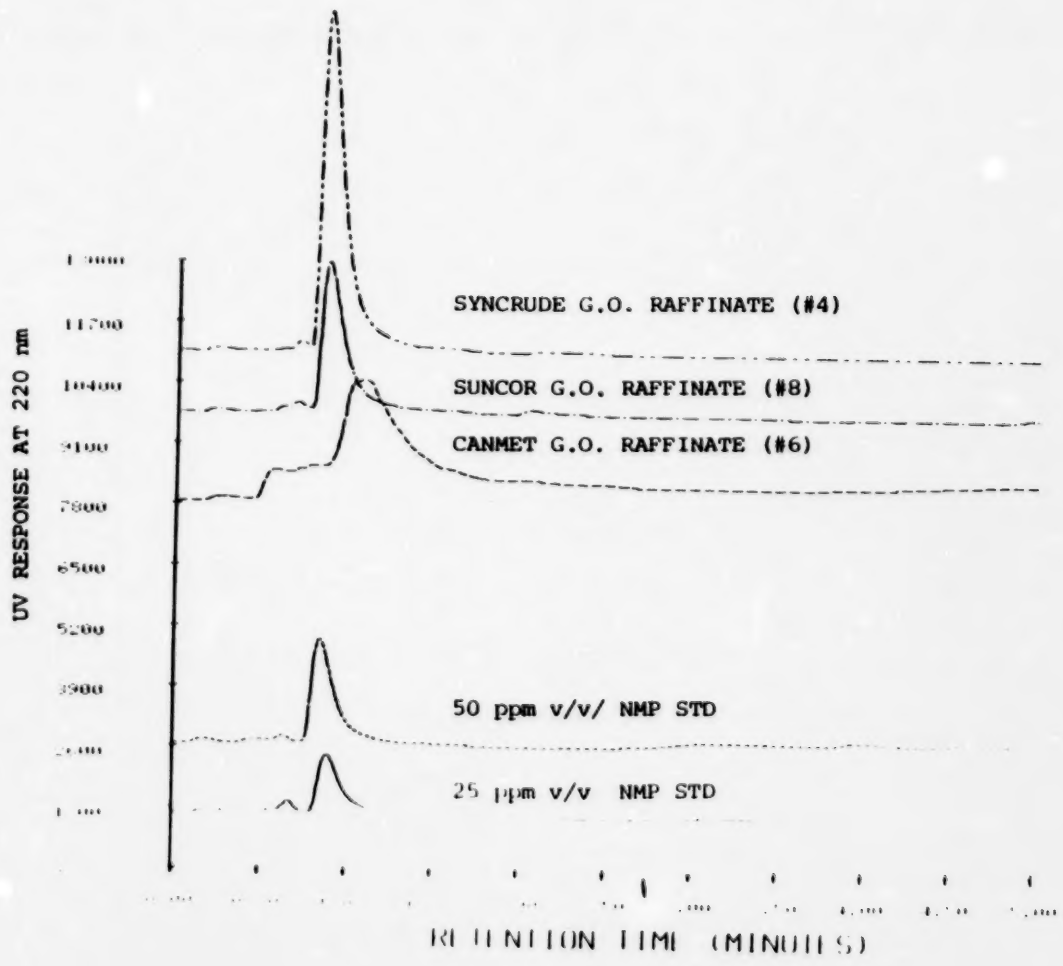
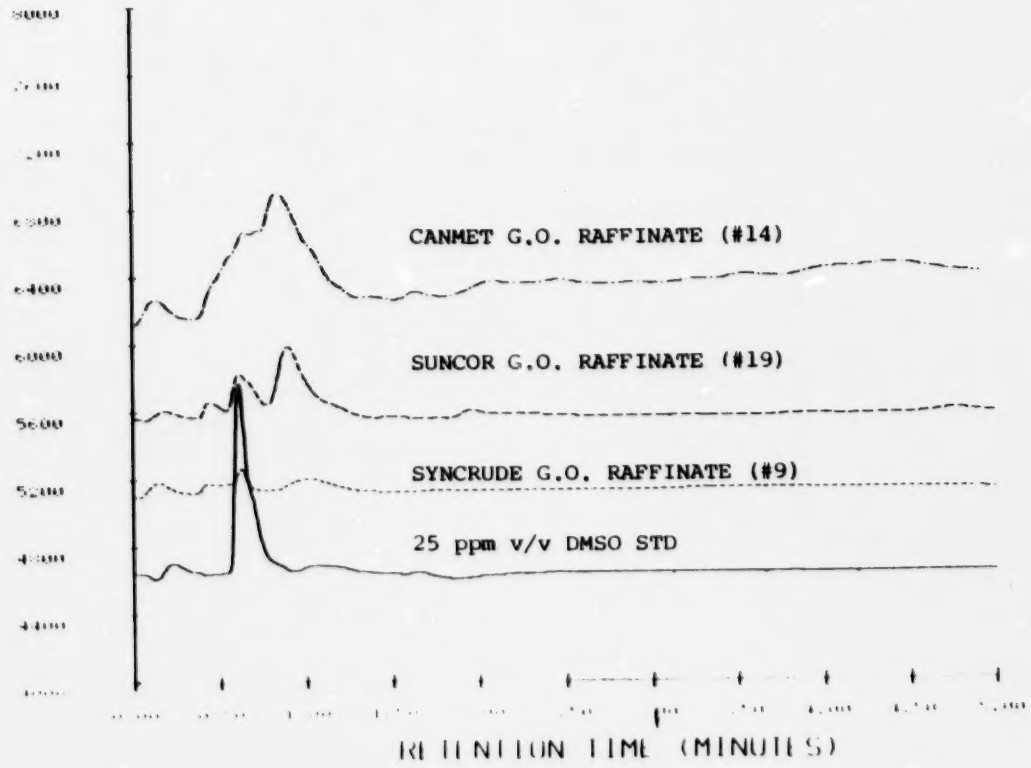

SYNCRUDE G.O. RAFFINATE (#4)
SUNCOR G.O. RAFFINATE (#8)
CANMET G.O. RAFFINATE (#6)
50 ppm v/v/ NMP STD
25 ppm v/v NMP STD
UV RESPONSE AT 220 nm
RETENTION TIME (MINUTES)

FIGURE A-4h: HPLC CHROMATOGRAMS OF GAS OIL RAFFINATES FROM DMSO/H_2O EXTRACTION



APPENDUM B

SELECTIVE EXTRACTION OF NITROGEN CONSTITUENTS FROM FLUID CATALYTIC CRACKING FEEDSTOCKS

L. E. Curts

Work on this project was conducted under the auspices of Canada Centre for Mineral and Energy Technology, Energy, Mines and Resources Canada

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RESEARCH AND DEVELOPMENT CENTRE
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TABLE OF CONTENTS

		Page No.
1.	INTRODUCTION	1
	1.1 Objective	1
	1.2 Justification	1
2.	EXPERIMENTAL	1
	2.1 Feed Samples	1
	2.2 Static Extractions	2
	2.3 Characterization Methods	4
3.	RESULTS AND DISCUSSION	5
	3.1 Feed Samples	5
	3.2 Static Extractions	5
4.	CONCLUSIONS AND RECOMMENDATIONS	6
	4.1 Static Extractions	6

TABLES AND FIGURE

1. INTRODUCTION

1.1 Objective

The main objective of this project was to use aprotic dipolar solvents to optimally extract nitrogen compounds from catalytic cracking feedstocks and thereby improve the fluid catalytic cracking (FCC) processability.

1.2 Justification

When synthetic crude oils become more economically attractive, their use will be primarily in the production of liquid transportation fuels. These fuels are currently produced by converting residual oils into naphtha and middle distillate using catalytic cracking.

Contract 15SQ.23440-3-9057 demonstrated the use of aprotic dipolar solvents to selectively remove nitrogen species from catalytic cracking feedstocks and improve light liquid product yields. This project was designed to compliment the work done in the earlier contract and investigate the following questions:

1. Is extraction applicable to other types of feed?

2. What is the effectiveness of alternate aprotic dipolar solvents on denitrogenation and FCC product yield?

3. What are the optimum solvent compositions and extraction conditions for maximum nitrogen removal?

4. What are the compositional characteristics of the extracted nitrogen compounds and those remaining in the raffinates?

2. EXPERIMENTAL

2.1 Feed Samples

Four feed samples were prepared at the R&D pilot plant facilities for this project. These samples consisted of two propane deasphalted (PDA) oils of different viscosities and a light and heavy fraction of raw Syncrude gas oil. The PDA oils were obtained from

IPL vacuum tower bottoms using a 6 ft. pilot plant propane deasphalting tower, which is a separation unit that combines extraction and precipitation. Liquid propane is used to dissolve or extract the low molecular weight paraffinic constituent and precipitate the high molecular weight aromatic asphalts present in vacuum tower bottoms. The degree of asphalt removal is controlled by operating temperature and propane-to-feed ratio.

The PDA unit was operated under two different severity conditions, the lower severity giving a higher oil yield product (36% wt) with increased viscosity (42 cSt @ 100°C) and the higher severity giving a lower oil yield product (21% wt) with reduced viscosity (30 cSt @ 100°C).

The raw Syncrude gas oil was cut at 385°C by batch distillation giving an IBP-385°C fraction and 385°C+ fraction.

The physical properties and compositional data for the feed samples are given in Tables 1 and 2.

2.2 Static Extractions

The four feed samples were extracted with N-Methylpyrrolidinone (NMP) based- and dimethylformamide (DMF) based-phases containing 10, 30 and 50 percent water.

The NMP was freshly distilled weekly to remove decomposition residue. The samples were extracted with equal volumes of the solvent mixtures and agitated in glass bottles for 1 hour.

The Syncrude feed samples were extracted at room temperature, however the PDA samples were waxy solids at room temperature and required heating to 80°C (approx. melting point). A heated water bath shaker was used with the PDA samples but in future an electrically heated shaker would be more suitable.

After solvent extraction, the feed samples were transferred to separatory funnels and heated for 24 hours at 90°C to reduce the emulsion.

The 385°C+ Syncrude sample formed an extremely stable emulsion that required salting with 50 ppm NaCl to reduce. All extracted samples were water washed for 1 hour in the mechanical shaker, then heated to 90°C for 24 hours to reduce emulsions. The extracted samples plus remaining emulsions were transferred to a Rotary Evaporation to remove all water from the raffinates.

Attempts to remove the water residue resulted in severe bumping of both PDA oils and the 385°C+ Syncrude feed sample. Samples required up to 10 hours of careful monitoring to safely remove residual water. Many raffinates were lost through bumping and required complete re-extraction, which used large quantities of time and samples.

Some nitrogen analyses of successful raffinates (Table 3) showed increased nitrogen levels indicating residual solvent contamination of the raffinates.

Insufficient raffinate remained for a second water washing and all samples required complete re-extraction, with two 1-h water washing stages.

At this point, an alternate method was developed to remove the residual water from the raffinate. This method uses the Dean & Stark apparatus (see Fig. 1) with toluene to remove the residual water safely. The procedure developed is as follows:

- Agitate 100 mL of sample with 100 mL of extraction mixture for 1 hour.
- Heat sample to 90°C for 24 h to reduce emulsion and drain.
- Add 50 mL toluene to raffinate.
- Water wash with 100 mL for 1 h and drain.
- Repeat water wash step and drain.
- Use Dean & Stark apparatus to remove residual water.
- Use Rotary Evaporator to flash off toluene.
- Submit raffinate for testing.

This procedure was tried successfully on IBP-385°C Syncrude feed samples.

An attempt was also made to improve the handling characteristics of PDA samples by creating a slurry with a 50/50 mixture in toluene which would also aid in water removal.

At this point in the project, approximately 5 of the original 7 litres of each processed feed sample had been used.

2.3 Characterization Methods

Mass Spectrometry

Mass spec analyses were performed on all samples using the Kratos Model 902 high resolution instrument.

Determination of Trace Metals

Routine atomic absorption was used for determining trace metals. Samples were reacted with dodecylbenzene sulfonic acid and ashed. The residue was dissolved in dilute HCl and then analyzed in a nitrous oxide-acetylene flame.

Determination of Sulphur Content

Routine method based on ASTM D1552 was employed using a LECO analyzer.

Determination of Nitrogen Content

Nitrogen contents on Syncrude samples and raffinate were measured by a chemiluminescence technique using an Antek Nitrogen Detector. The PDA samples required analyses by the Kjeldhal method (ASTM D3228) which has proven to be more accurate on heavier hydrocarbons.

Infrared Compositional Analysis

Solutions of approximately 1 g of sample in 50 mL of CCl_4 were prepared. The IR spectra of feeds were taken using the dispersive IR spectrometer (Perkin Elmer 283B). The pyrrolic NH and alcoholic -OH content were determined using carbazole and sitosterol as calibration standards.

Determination of Distillation Range

Distillation ranges for the four feed samples were determined chromatographically using a Hewlett Packard 5880A gas chromatograph. The procedure is a modification of the ASTM D2887 method.

3. RESULTS AND DISCUSSION

3.1 Feed Samples

The physical characteristics of the four feed samples are given in Tables 1 and 2. The low yield PDA oil has been more severely deasphalted as shown in the higher aniline point (i.e. low aromatics), lower viscosity, and lower percentage of heavy aromatics by mass spectrometry. The high yield PDA oil contains more residual asphalt.

Note the 25% reduction in basic nitrogen level by increasing PDA unit severity.

The Syncrude feed samples show approximately 80% of the total nitrogen content is contained in the 385°C+ fraction. Compositionally the 385°C+ feed sample is similar to that used in contract 15SQ.23440-3-9057.

3.2 Static Extraction

The objective of the static experiments was to determine the optimum solvent/water ratio (extraction mixture) for maximum extraction of nitrogen compounds from each feed sample. Once the optimum extraction mixture was established then the most effective extraction mixture to feed sample ratio was to be determined.

Nitrogen results on the extracted feed samples given in Table 3 show a single water wash step did not effectively remove residual solvent and that two or more water washings would be required to remove all traces of extraction solvent. With increased water washing, reduced recovery of raffinate would also be expected, perhaps negating any advantage in FCC feedstock upgrading.

As discussed in the Experimental Section 2.2, this preliminary work in the contract was plagued with methodology problems that used significant amounts of the processed feed samples and time.

Since the remainder of the contract involved an extensive number of multistage and static extractions, it was felt that further work on the contract could not proceed until a method was developed to extract the feed samples safely and consistently.

The method given in Experimental Section 2.2 was found to work successfully; however, due to a manpower shortage and the need to process four new feed samples to complete the work, the contract is on hold pending review of time and available manpower. The amount of time required to complete this project substantially exceeds that which remains in the contract.

4. CONCLUSIONS AND RECOMMENDATIONS

4.1 Static Extractions

A method has now been developed to technically allow work to proceed on this contract.

Insufficient processed feed remains to repeat the contract as written.

The estimated cost of reprocessing 10 litres of all four feed samples would be at least 150 manhours and the additional effort to complete the contract as written would total 900 manhours.

To repeat any work would require designing and building an electrically heated shaker capable of holding approximately 10 separatory funnel type extraction vessels.

Our recommendation, in light of the current and projected crude oil prices, is to discontinue this program.

TABLE B-1

PDA AND SYNCRUDE FEEDSTOCK CHARACTERIZATION

Characteristics	Feed			
	PDA Oil Low Yield	PDA Oil High Yield	Syncrude Gas Oil IBP-385°C	Syncrude Gas Oil 385°C+
Specific Gravity @ 60°F (D4052)	0.9063	0.9142	0.9120	0.9521
Aniline Pt., °C (D611)	115	109	45.8	64.4
Viscosity (D445)				
cSt @ 40°C	NA*	NA*	8.31	189.65
cSt @ 100°C	30.00	42.71	2.14	11.34
Conradson Carbon, % (D189)	1.75	2.36	0.009	0.22
Bromine No.	NA**	NA**	0.71	NA**
Sulphur Content, % (D2622)	0.58	0.51	0.20	0.44
Total Nitrogen, ppm (D3431)	980***	1024***	460	1616
Basic Nitrogen, ppm (D3431)	445***	590***	113	250
Metals Content, g/g				
Ni	0.25	0.30	0.06	0.09
V	0.14	0.23	0.14	0.10
Fe	0.29	0.68	0.69	4.75
Cu	0.54	<0.05	<0.05	0.10
Normal Boiling Range, °C (Gas Chromatography)				
IBP	338	380	187	360
5%	463	484	234	372
10%	491	508	256	378
50%	564	577	327	419
90%	618	629	365	485
FBP	649+	649+	384	571
Infrared Compositional Analysis				
Carbazole N-H, ppm	215	233	144	717
Aromatic O-H, %	0.4	0.3	<0.1	><0.1

* PDA oils waxy solids at 40°C

** 90% distillation >316°C

*** Kjeldhal analysis (D322)

TABLE B-2

HIGH RESOLUTION MASS SPECTROMETRY ANALYSES OF PDA AND SYNCRUDE FEEDSTOCKS

Component Types	PDA Oil Low Yield	PDA Oil High Yield	Syncrude Gas Oil IBP-385°C	Syncrude Gas Oil 385°C+
Paraffins	14.5	13.0	5.4	3.1
Cycloparaffins	53.9	46.8	40.0	37.8
Monoaromatics	14.5	13.1	34.3	21.0
Diaromatics	7.9	9.2	15.3	18.1
Triaromatics	3.5	4.9	3.3	10.7
Tetraaromatics	1.6	3.8	0.6	4.6
Pentaaromatics	1.5	3.5	0.1	2.7
Polyaromatics	0.1	1.6	0.0	0.0
Thiophenes	2.5	3.8	1.0	2.0
Furans	0.1	0.4	0.0	0.0

TABLE B-3

TOTAL NITROGEN RESULTS (ppm)
After Single Water Wash Step

	PDA Oil Low Yield	PDA Oil High Yield	Syncrude Gas Oil IBP-385°C	Syncrude Gas Oil 385°C+
Feed	980	1024	460	1616
NMP/10% Water Extraction	NA	1180 R=95	208 (144)* R=91	1299 R=89
NMP/30% Water Extraction	930 R=97	1150 R=96	274 (246)* R=92	1669 R=95
NMP/50% Water Extraction	NA	1890 R=99	359 (319)* R=97	2162 R=97
DMF/10% Water Extraction	NA	1140 R=95	188 (164)* R=91	1202 R=91
DMF/30% Water Extraction	970 R=98	1170 R=96	354 (280)* R=93	1574 R=95
DMF/50% Water Extraction	930 R=99	1170 R=97	368 (352)* R=97	1687 R=94

NA - analyses not available due to bumping
R - % recovery after 1 water wash

Note: ()* Nitrogen results in brackets represent two water washings.